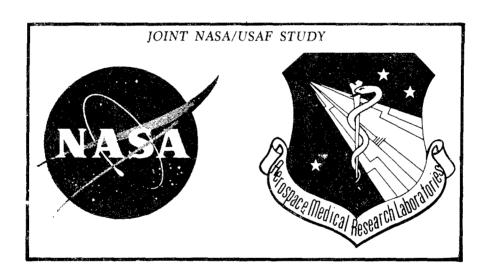
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IDENTIFICATION OF VOLATILE CONTAMINANTS OF SPACE CABIN MATERIALS

J. V. PUSTINGER, JR.

F. N. HODGSON



JULY 1968

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FOREWORD

The study was conducted at the Dayton Laboratory of the Monsanto Research Corporation, Dayton, Ohio, under Contract No. F33615-67-C-1357. The principal investigator was Mr. F. Neil Hodgson for the Monsanto Research Corporation. The study was started in January 1967 and was completed in February 1968. The entire study was under the project leadership of Mr. John V. Pustinger, Jr., of Monsanto Research Corporation.

This research was initiated by the Chemical Hazards Branch, Toxic Hazards Division, Biomedical Laboratory in support of Project 6302, "Toxic Hazards of Propellants and Materials," Task 630204, "Environmental Pollution." Mr. Solomon Brokeshoulder and Dr. Gerd A. Kleineberg of the Chemical Hazards Branch were contract monitors for the Aerospace Medical Research Laboratories.

The authors acknowledge the invaluable assistance of Mr. Bruce E. Boggs, Mr. Donald Q. Douglas, and Mr. John E. Strobel; all of Monsanto Research Corporation.

This technical report has been reviewed and is approved.

WAYNE H. McCANDLESS
Technical Director
Biomedical Laboratory
Aerospace Medical Research Laboratories

ABSTRACT

Fifty-three candidate materials for space cabin construction were tested to establish volatile gas-off and oxidation products. Testing was accomplished by two methods:

- (a) preliminary screening by thermogravimetric analysis to determine weight loss during 24 hours at 25°C to 68°C in a nitrogen atmosphere at 5 psia.
- (b) storage tests at 68°C for 72 hours and at 25°C for 30 and 60 days in oxygen at 5 psia, followed by analyses of the chamber gases.

The preliminary screening by measuring weight loss was to determine those materials which exhibit weight losses between 0.001% and 1.0%, exclusive of water. Materials falling within this range were studied further in storage tests to determine the nature of the individual components evolved from the candidate material. Those materials falling outside this range were conditionally excluded from further tests. Weight loss data, thermogravimetric curves, gas chromatograms of volatile contaminants, and the nature and quantities of individual components evolved from the candidate materials are reported.

In addition to the gas-off experiments, gas chromatographic and mass spectrometric analyses were performed on 7 samples of atmospheres from bio-environmental systems.

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SECTION I

INTRODUCTION

Previous material evaluation programs 1,2 have shown that certain candidate space cabin materials, when tested under simulated space cabin environmental conditions (5 psia oxygen or air at 25°C or 68°C), yield an abundance of volatile contaminants to the atmosphere. In these programs, storage systems and analytical methods had been devised to identify and to measure quantitatively the gas-off products in the gas phase. As a continuation of these studies, an evaluation program, based on weight-loss measurements and on the storage tests and attendant analyses used previously, has been developed for screening 53 candidate space cabin materials.

Storage tests and subsequent analyses of the gases in the chamber atmosphere are time-consuming. An initial screening of the materials is desirable to discover the materials that give an abundance of volatile contaminants, and those that yield practically none. On this basis, materials could be selected for further testing or conditionally eliminated from additional tests. Materials giving moderate amounts of contamination would be tested further to identify and to measure individual contaminants.

The merit in the use of weight-loss measurements is the comparative simplicity of the equipment and the relatively short measurement time. The intrinsic limitation of this approach is the inability to identify directly the evolved products. On the other hand, the advantage of closed chamber storage tests is the ease with which identification and quantitative analyses of the head gases can be performed; however, the disadvantages are the time-consuming processes of storing specimens for long periods and of performing the identifications of the volatiles. Our program was developed to take advantage of the best characteristics of both approaches.

Pustinger, J. V., Hodgson, F. N., Ross, W. D., 1966, Identification of Volatile Contaminants of Space Cabin Materials, AMRL-TR-66-53, Aerospace Medical Research Laboratories, Wright-Patterson Air Force Base, Ohio, pp. XVI + 194.

²Pustinger, J. V., Hodgson, F. N., 1967, Identification of Volatile Contaminants of Space Cabin Materials, AMRL-TR-67-58, Aerospace Medical Research Laboratories, Wright-Patterson Air Force Base, Ohio, pp. XVI + 194

The initial screening procedure, using thermogravimetric techniques for measuring weight loss at moderate temperatures (ambient to $68 \pm 2^{\circ}\text{C}$) for 24 hours in 5 psia nitrogen, was developed to select those candidate materials that lose from 0.001 to 1.0% of their weight, excluding water. The selected materials are then stored in 9-liter chambers at $68 \pm 2^{\circ}\text{C}$ for 72 hours and at 25 \pm 2°C for periods of 30 and 60 days. Atmosphere in the chambers is oxygen at a pressure of 5 psia. The gaseous contaminants evolved from the test materials are identified by combinations of gas chromatography and mass spectrometry.

SECTION II

GAS-OFF EXPERIMENTS

A. EXPERIMENTAL METHOD

1. Types of Candidate Materials and Sample Preparation

Table I lists the candidate materials for cabin construction used in these experiments; all materials tested are commercial products provided by the Government. Whenever possible, the candidate materials were studied in the same state as received. Materials such as paints and inks were applied to an aluminum foil substrate and then dried at the designated temperature and for the designated time according to the manufacturer's or the Air Force's directions. Similarly, two-part resins were mixed and cured according to procedures provided by the manufacturers. All calculations were made on the basis of dry sample weight.

Specimens used for thermogravimetric analysis (TGA) were conditioned at 23°C in a desiccator over phosphorus pentoxide for 24 hours prior to testing. For storage tests at 72 hours, 30 days, and 60 days, no pretreatment of samples was performed beyond the curing procedures cited by the manufacturers or the Air Force. The procedure for preconditioning the TGA specimens was devised to minimize adsorbed water and to put all samples on the same basis for comparing relative weight loss.

For storage tests for 72 hours, 30 days, and 60 days, a weighed portion of each sample was placed into a 9-liter chamber in a manner to expose the largest possible surface area. Generally, approximately 10-gram specimens were used; however, in cases where less sample was available, or when the bulk volume of the sample was excessively large, smaller specimens were used. When the bulk volume was too large and subdividing was necessary, freshly exposed surfaces were further cured at ambient conditions, i.e., 23°C and atmospheric air pressure, for 30 days or a minimum of 14 days.

Individual specimens of each candidate material were contained in 9-liter, borosilicate glass chambers for periods of 30 and 60 days at $25 \pm 3^{\circ}$ C, and for a period of 72 hours at $68 \pm 2^{\circ}$ C, under an oxygen atmosphere at 5 psia and 20-40% relative humidity. The chamber design and pretreatment of the chambers were the same as reported earlier. Two-hundred chambers were used (on a staggered schedule) over a span of 13 months to permit analyses to be performed after 72 hours,

¹Pustinger, Hodgson, and Ross, p.1.

Table I

SPACE CABIN TEST MATERIALS

Code No.	Material	Code No.	Material
	Schjeldahl (Mylar) X850	AF 209	Irradiated Polyolefin,
DAC 003	ETV 731 Stiteone		Myler Dolvester
	Saran Wrap - Tvpe 18		Trrediated Polyclefia Thermofit BNR
	Aclar Type 33-C	AF 302	Eccobond 700 Adhesive
	Shonka A-150 Polyolefin/Polyamide		Thermonlastic Coating.
	Spandex Lycra Polyurethane		1169 A/B Coating
			Stvogst 1090 Cat-1
	RIV 615	AF 403	Stvcast 2651-50 CAF 1
	Stycast 2651/Catalyst II (Epoxy)		S111cone RTV 521
	Hathone HA 7236		Adiorene L-100 MCC Adhesive
	Butyl Rubber 00996-33L		Silicone RTV 502 Rubber
	Polyurethane 00996-39B		S111cone 030079
	Silicone 19513-10f		Silicone RTV 503
	Boltaron ABS Polymer		Tape, Stycast 2651-Cat 9
			O11. Aero Shell #7
	-		Paint, Lacquer Stik White
	-		Conductive Paint
	Bondmaster E611 (Modified Epoxy)		Resin Emerlon 310
	Epoxy-Polyamide, V-9 Silver Epon 815		Varnish E44
	Silicone EMS 323		Polvurethane PC-18
	Viton A EMS 338 (Fluoro Elastomer)		Ink F-150
	er EMS 342		Ink #41 Black
	Silicone Rubber EMS 345		Ink Red Marking VF-200
	Hapalon Rubber EMS 355		Nylon Cond Style 18
	Urethane EMS 366	AF 552	Nylon Cord Style 21
	Polyester (Dacron) Silicone		Lastic 60,000 RF1
	Eboxy Primer M-602		t !!

30 and 60 days. Control chambers (containing only aluminum foil) were processed concurrently with those chambers containing the test materials. No contamination was detected from the control chambers.

2. Preparation of Chamber Atmospheres

After each specimen was inserted into the 9-liter chamber, the test chamber was filled to a pressure of one atmosphere with oxygen, saturated with water vapor. The gas was saturated with water by bubbling 99.5% oxygen (conforming to Type I of MIL-0-27210) through triply distilled water at 23°C. Test conditions were attained by subsequently reducing the pressure in the chamber to 5 psia, resulting in a test atmosphere of oxygen at 5 psia with a relative humidity of approximately 33%.

Measurement of relative humidity was made with an Alnor Type 7300 Dew-Pointer (Illinois Testing Laboratories, Inc., Chicago, Ill.).

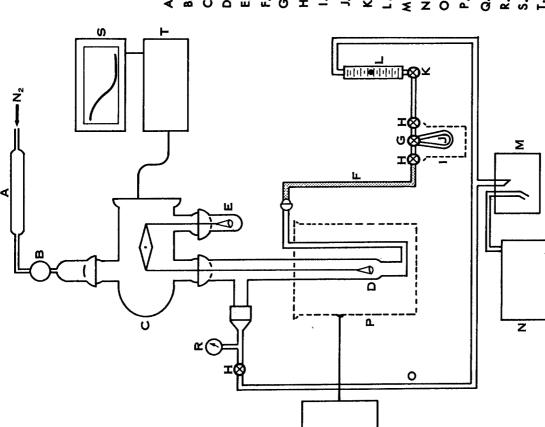
Test atmospheres were maintained at $25 \pm 3^{\circ}\text{C}$ by storing chambers in a temperature-controlled room for periods of 30 and 60 days. The chambers that were tested at $68 \pm 2^{\circ}\text{C}$ were stored in a constant-temperature cabinet (Blue M Electric Co., Stabil-Therm DL132C).

3. Analytical Methods

a. Weight Loss Measurements

Conditional screening of candidate materials was performed by measuring the weight loss of the material using thermogravimetric measurements (TGA). Weight loss from approximately 10 g of a material was recorded continuously as the temperature of its environment was raised from ambient (approximately 23°C) to $68 \pm 1^{\circ}\text{C}$ in 4 hours and then maintained at $68 \pm 1^{\circ}\text{C}$ for 20 hours or until weight remained constant for 2 hours. All TGA measurements were made in dried, prepurified nitrogen at 5 psia.

Thermogravimetric measurements were made with a Cahn RH Electrobalance equipped with a modified F&M Model 240-00 Power Proportioning Temperature Programmer, Flo-Thru tube, a temperature programmed oil bath, and a 1 mv recorder (Figures 1, 2, and 3). The Cahn RH Electrobalance is a vacuum- and controlled-atmosphere automatic recording balance with 100-gram capacity



SCREENING APPARATUS **THERMOGRAVIMETRIC**

A. Drierite and P2O5 Drying Tube

B. Matheson Absolute Pressure Regulator

C. Cahn Electrobalance

D. Sample

E. Tare Weight

F. Calcium Carbide Reactor Tube

G Gas Sampling Valve

H. Toggle Valves

I.Dewar Flask with Liquid N₂

J. Sampling Loop

K. Needle Vaive L.Flowmeter

M.Pump Isolation Trap

N.Pump O. Auxiliary Evacuation Line

P.Oil Bath

Q. Temperature Programmer

R.Pressure Gauge

S.Strip Chart Recorder

T.Balance Control Unit

Schematic of Thermogravimetric Screening Apparatus. Figure 1.

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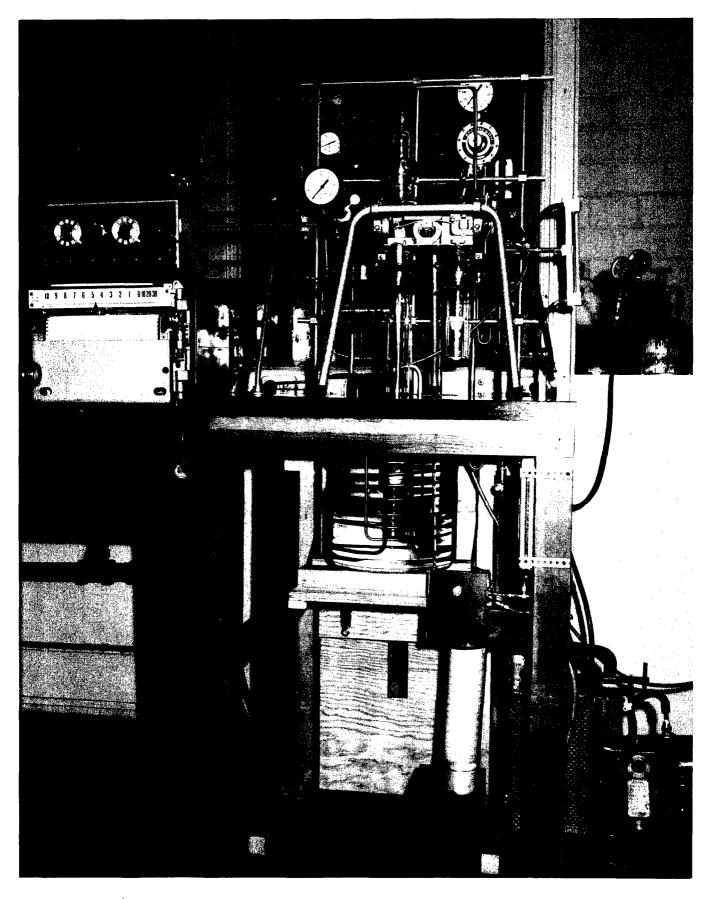


Figure 2. Cahn Electrobalance in TGA System.

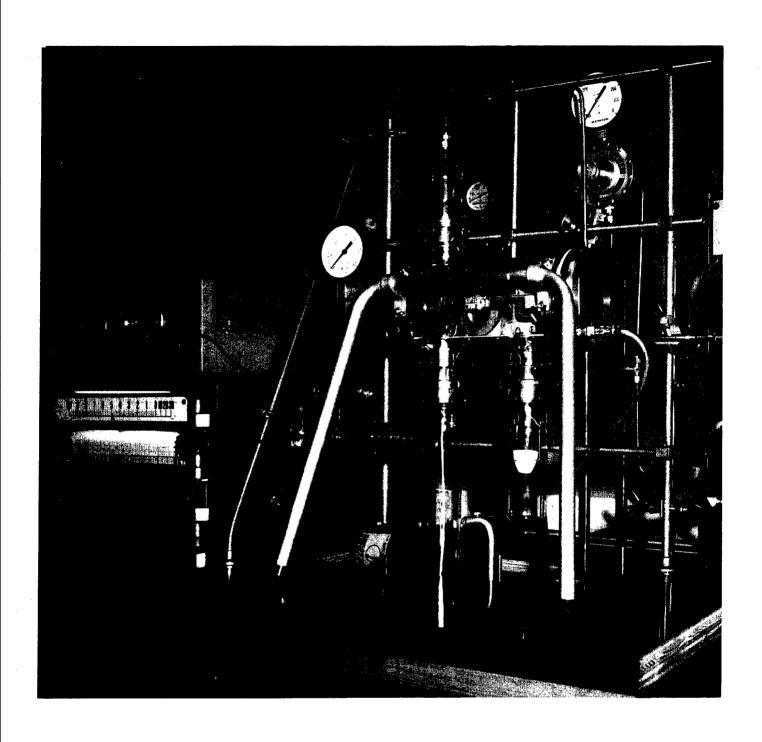


Figure 3. Complete TGA System.

and a sensitivity of 10^{-6} g. This thermogravimetric system will permit the handling of relatively large specimens, 1-10 grams, and the detection of small weight changes, a 10^{-7} fraction of the load.

The arrangement of the components of the TGA system is shown in Figures 1, 2, and 3. To permit greater control and more uniform heating, the sample is heated with an oil bath rather than an oven or furnace. The slow heating rate of one degree every six minutes is more easily achieved by adjusting the temperature of the oil bath than by using a heating unit directly. The temperature was programmed with an F&M Power Proportioning Temperature Programmer that had been modified to permit the slow heating rate.

Water evolved from the sample was monitored by either of two gas chromatographic methods:

- (a) by converting the water to acetylene by a reaction with calcium carbide and analyzing for acetylene;
- (b) by cryogenically trapping the desorbed water and subsequently performing a direct measurement for water.

Gas chromatographic analyses for acetylene were performed with a flame ionization detector (F&M 1609) and a Porapak T column, whereas direct gas chromatographic measurements for water were performed with a thermal conductivity detector (Barber-Colman 61C) and a Porapak T column.

b. Gas Chromatographic Analysis of Gas-Off Products

Carbon monoxide, methane, and gas chromatographic analyses were performed by techniques reported earlier^{1,2}. All atmospheres in the test chamber were sampled for analysis at the temperature of the test, i.e., 25°C or 68°C.

The general analyses of the gas-off products by gas chromatography were performed on an F&M Model 810 Research Gas Chromatograph equipped with dual flame ionization detectors. A general purpose column, 20-ft x 0.25-in. ss., 20% Triton X-305 on 60/80 mesh Gas Chrom Z was used for most samples. Gas-off products from four samples (DAC 006, DAC 017, DAC 018, and DAC 020), tested in the first quarter of the program, were analyzed by means of a double column, 20-ft x 0.25-in. ss., 5% Carbowax 20M on 60/80 mesh Gas-Pack F with a pre-column of 12-ft x 1/8-in. ss., 7% neopentylglycolsuccinate on 60/80 mesh Gas Pack F. used

²Pustinger and Hodgson, p.1.

Pustinger, Hodgson, and Ross, p.1.

earlier². The advantage of using the Triton X-305 column is better separation of early eluting components.

Quantitative gas chromatography data were obtained by comparing the peak heights with those of a standard mixture. Gas chromatographic instrument conditions are presented in Appendix III. Table XLV.

Identifications of gas chromatographic components were made by mass spectrometric analysis of the gas chromatographic effluent. In most cases component identification was accomplished by the direct, tandem coupling of a fast scan mass spectrometer, CEC 21-104, to the gas chromatograph. By splitting the effluent, a portion was directed to the flame ionization detector and a second portion was introduced directly into the mass spectrometer. With some samples, a concentration step requiring the cryogenic trapping of the major portion of the total 9-liter volume was necessary. This condensate was subsequently separated into its components and characterized by the coupled gas chromatograph-mass spectrometer system.

c. Mass Spectrometric Analysis of Gas-Off Products

Two types of mass spectrometric analyses were performed for each sample. A composite analysis of the atmosphere of each 9-liter bottle was made on an aliquot (125 cc) of the atmosphere with a Consolidated Electrodynamics Corporation Model 21-103C Mass Spectrometer. As indicated in Section II-A-3-b, a fast scan Consolidated Electrodynamics Corporation Model 21-104 Mass Spectrometer was used in a direct couple with a gas chromatograph to identify the components eluting from the chromatograph. Both approaches are necessary to insure complete characterization of the chamber atmospheres.

Identifications of individual components were made by mass spectrometry and were supported by infrared absorption and by gas chromatographic data as needed. Most of the mass spectra obtained were compared to API (American Petroleum Institute) reference spectra. In cases where the required mass spectrum does not appear in the API collection, comparison was made with spectra from our laboratory files or from the literature.

B. RESULTS AND DISCUSSION

Table II lists the types of compounds detected in the chamber atmospheres. These data represent compounds exclusive of H_2O_3 , O_2 , O_2 , and N_2 .

²Pustinger and Hodgson, p.1

¹Pustinger, Hodgson and Ross, p.1

Table II

TYPES OF COMPOUNDS DETECTED

Inorganics

Carbon Monoxide

II. Alkanes

C4 Hydrocarbon(s) C5 Hydrocarbon(s) C6 Hydrocarbon(s) Methane Propane

III. Alkenes

Ethylene
C. Unsaturated Hydrocarbons
Di-isobutylene
Isobutylene
Propylene
Tetra-isobutylene
Trichloroethylene
Tri-isobutylene
Tri-isobutylene

IV. Alcohols

n-Butanol
2-Butanol
tert-Butanol
2-Buten-1-ol
2-n-Butoxyethanol
Cyclohexanol
Ethanol
2-Ethoxyethanol
Isopropanol
Methanol
3-Methyl-1-butanol
3-Methyl-1-propanol
2-Phenyl-2-propanol
n-Propanol
2-n-Propoxyethanol

V. Alkyl Halides

Chloroform
Homologous Series of
Chloro-fluorocarbons
Trichloromonofluoromethane

VI. Carboxylic Acids and Their Derivatives

Acetic Acid
2-n-Butoxyethylacetate
Butylacetate
2-Ethoxyethylacetate
Ethylformate
Formic Acid
Methylformate
Propylacetate

VII. Aldehydes

Acetaldehyde Butyraldehyde Formaldehyde Propionaldehyde Valeraldehyde

VIII. Ketones

Acetone
Acetophenone
2-Butanone (Methylethylketone)
Cyclopentanone
Di-isopropylketone
Di-n-propylketone
Hexanone
4-Methyl-2-pentanone
(Methylisobutylketone)
Pentanone

IX. Ethers

1,3-Dioxane Propylene Oxide N-Methylmorpholine

X. Aromatic Hydrocarbons

Benzene
C3 Alkylbenzenes
C4 Alkylbenzenes
Ethylbenzene
Methylstyrene
Naphthalene
Styrene
Toluene
Xylenes

XI. Aromatic Hydroxy Compounds

Phenol

XII. Silicon Compounds

Various Cyclic and Linear Methylsiloxane Polymers Materials that showed no products (exclusive of H_2O , CO_2 , O_2 , and N_2) are listed in Table III.

Materials showing weight loss in excess of 1% and for which no analytical data were obtained are listed in Table IV. Although these materials were conditionally screened and eliminated from further testing based on TGA measurements, all candidate materials in this program were processed in storage tests for 72 hours, 30 days, and 60 days, and their gas-off products were analyzed with gas chromatography and mass spectrometry to confirm the results of the TGA measurements. In most cases, where the weight loss exceeded 1%, gas chromatographic and mass spectrometric data were obtained, only to establish the magnitude of the total off-gases. No specific identifications or measurement of quantities of individual components have been made. Others were analyzed completely for comparison purposes with the TGA data.

Analytical data are presented in Appendix I, Table V, Figures 4 to 56 (Thermogravimetric Weight Loss Data and TGA Curves); Appendix II, Tables VI to LXIV (Analytical Results for Gas-Off Experiments); and Appendix III, Figures 57 to 95 (Gas Chromatograms for Gas-Off Experiments).

All values appearing in the tables of Appendices I and II are calculated on the basis of the dried or cured samples (this is important in the case of paints and coatings where the weight of the material is substantially reduced by drying).

Some gas-off products are identified by compound type only, e.g., alkylbenzene(s), C4 alkylbenzene(s), or C4 hydrocarbon. In these cases several homologues or isomers may be present; however, they have not been identified individually.

Some of the gas-off products from silicone base materials were also calculated collectively. These were the volatile linear and cyclic siloxane polymers (having dimethyl siloxy groups as monomer units), which had been observed in previous gas-off studies^{1,2}. Although separate peaks are noted in the gas chromatograms (Appendix III), these volatile silicones are listed collectively in the tables of gas-off data (Appendix II) as silicone oil.

Although no distinct correlation of composition and the shape of the TGA curve can be derived from the limited data, some similarities in TGA curves are noted, e.g., two materials containing polyamide [DAC 007, Shonka A-150 Polyolefin/Polyamide (Figure 9) and AF 054, Epoxy-Polyamide, V-9 Silver Spon 815 (Figure 23)]. Also, certain similarities in the total weight loss data are observed for materials of a particular type, e.g.,

²Pustinger and Hodgson, p.1

Pustinger, Hodgson and Ross, p.1.

Table III

CANDIDATE MATERIALS EXHIBITING NO GAS-OFF PRODUCTS

- DAC 001 Mylar, Schjeldahl X850
- DAC 003 Silicone, RTV 731
- DAC 005 Saran Wrap, Type 18
- DAC 008 Polyurethane, Spandex Lycra
- DAC 011 Polyurethane, PR 1535
- DAC 012 Silicone, RTV 615
- DAC 013 Epoxy, Stycast 2651/Catalyst II
- DAC 022 Polycarbonate, Merlon 1000
 - AF 064 Fluoroelastomer, Viton A, EMS 338
 - AF 561 Fabric, Lastic 60,000 RF1

Table IV

CANDIDATE MATERIALS CONDITIONALLY WITHDRAWN FROM FURTHER TESTING

(Based on Excessive Weight Loss and Preliminary Gas Chromatographic and Mass Spectrometric Measurements)

AF 325 - Thermoplastic Coating, 1169 A/B

AF 533 - Polyurethane, PC-18 Coating

AF 536 - Ink, F-150 Marking

AF 537 - Ink, #41 Black

AF 540 - Ink, VF-200 Red Marking

AF 063 (Silicone Rubber EMS 323), AF 065 (Silicone Rubber EMS 342), and AF 066 (Silicone Rubber EMS 345) lost 0.25%, 0.26%, and 0.26% of their weight, respectively. However, the significance of this observation is not clear since other, non-silicone materials, also exhibit weight loss of similar proportions.

The need for gas chromatographic and mass spectrometric confirmatory evidence in the screening of the candidate materials by TGA measurement results from the inability to measure quantitatively the amount of water evolved during the TGA measurement. The reliability of the two gas chromatographic methods for water, i.e., (a) conversion to acetylene and subsequent acetylene analysis, and (b) direct analysis for water, was established through the use of known water standards. However, difficulty was encountered when attempting to distinguish the water evolved from the sample, from that present in the pre-dried, pre-purified nitrogen used as the atmosphere and from that adsorbed on the inner surface of the TGA apparatus.

Although the amount of water present in the apparatus is very small, apparently there are sufficient quantities adsorbed on the glass at 23 to 68°C to yield a relatively large amount into the gas phase during a 24-hour test. Although not conclusive, an apparent adsorption-desorption equilibrium is established with the sample, metal and glass surfaces of the apparatus, and the gas phase, such that a relatively constant amount of water (20-28 mg) is isolated during a 24-hour test. Experiments performed without sample to establish the background level of water yielded similar quantities of water. To eliminate this problem, the apparatus is being modified to provide a direct measure of water desorbed from the sample by locating a hygrometer probe at the sample site.

A comparison of weight loss data (23 to 68°C) with the quantitative analyses of volatiles from storage tests at 68°C should be made with care. Several opposing effects should be noted. A large number of materials continue to desorb considerable quantities of volatiles after 24 hours. Thus, more gas-off products should be present in the atmosphere during 72-hour tests than gas-off products detected as weight loss during 24-hour TGA measurements. However, adsorption of volatiles on chamber surfaces results in an opposing effect. Considerable quantities of polar and relatively non-volatile gas-off products are retained on the chamber surfaces in the storage tests even at 68°C. Oily films were deposited on chamber walls in many of the tests. This effect was particularly noticeable when testing silicones, in that, relatively low molecular weight silicone oil condensed on

the chamber surfaces. Similarly, considerable amounts of butoxy-ethylacetate (identified by infrared spectra) were collected on the surfaces of test chambers for AF 522.

Material No. DAC 014, Hathone HA 7236, a polymeric foam, showed an unusual behavior during the TGA measurements. Solid material in the form of a fine dust was lost from the crucible. This was perhaps due to the rupture of gas-filled bubbles resulting from the decreased pressure (5 psia). Collection of the dust for weighing was difficult due to the static charge associated with this type of material, but it was estimated to be approximately 2-3 mgs for a 2-g specimen. The formation of this dust could perhaps, in itself, be considered a significant atmospheric contaminant.

SECTION III

BIO-ENVIRONMENTAL AND SPECIAL ANALYSES

A special study was performed to determine the gas-off products of Hetron Polyester Resin. This material is not a candidate space cabin material, but it is of interest to the Government as a construction material for terrestrial use. Ten grams of the material was stored in a 9-liter gas-off chamber for 30 days in an air atmosphere at a pressure of 1 atmosphere at 25°C. The most abundant product observed in the off-gases was methylethyl-ketone. This compound undoubtedly arises from methylethylketone peroxide used in the formulation of the polymer. However, due to lack of data on the stability of MEK peroxide, it has not been established whether the peroxide decomposes during storage or during analysis. Table XLVI lists the gas-off products from this material.

Special analyses Nos. 2 and 3 were performed on air specimens collected on 28 March 1967 from an Air Force test chamber and from the room air immediately outside the chamber. Data from these analyses are shown in Table XLVII.

Special analyses Nos. 4, 5, 6 and 7 were performed on bioenvironmental specimens contained in four 50-liter cylinders which were received from the Air Force on 25 August 1967. The results, given in Table XLVIII, are exclusive of water and carbon dioxide. It will be noted that low molecular weight chlorinated fluorocarbons are present in cylinder #1 (sample No. 4). These appear to be similar to the volatiles which have been observed to arise from certain fluorocarbon lubricants.

SECTION IV

CONCLUSIONS AND RECOMMENDATIONS

Considerable differences in levels of volatiles as determined from weight-loss measurements (TGA) and from storage tests and analyses of atmosphere of the storage chambers are observed. A major contributing factor is the adsorption of volatiles on the chamber walls.

The application of thermogravimetric measurements for screening candidate materials has proven useful, but a more direct measurement of water at the sample site must be made to provide more reliable data. We recommend the use of a hygrometer probe at the sample site. Such a device is being incorporated in a modification of the TGA system used in this test program. Until a reliable direct water analysis can be performed, we recommend continued 72 hours storage tests at 68°C and attendant atmosphere analyses to confirm the level of volatile products desorbed from the candidate materials.

APPENDIX I

THERMOGRAVIMETRIC PATTERNS OF CANDIDATE SPACE CABIN MATERIALS

The thermogravimetric analysis (TGA) patterns shown in this appendix were obtained on a Cahn RH Electrobalance. Comparison of the weight loss patterns should be made with care since varying amounts of sample were used to obtain the TGA patterns. The quantity of material used for each TGA measurement is shown on the reproduced pattern.

TGA curves appear in order of their Air Force serial numbers. Names of materials are those submitted by the Air Force.

Table V

TOTAL WEIGHT LOSS AS DETERMINED BY THERMOGRAVIMETRIC ANALYSIS OF CANDIDATE MATERIALS

Weight Loss (%)	00000000000000000000000000000000000000
Weight Loss (mg)	######################################
Sample Weight (g)	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
Code No.	AAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAA
Weight Loss (%)	
Weight Loss (mg)	111 0 0 0 111 1 0 0 1110 101000
Sample Weight (g)	
Code No.	DDDDDDDDDDDDDDDDDDDDDDDDDDDDDDDDDDDDDD

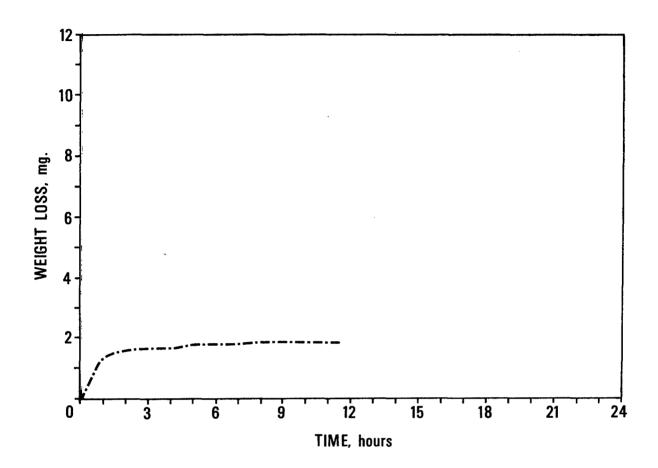


Figure 4. TGA Curve of Schjedahl (Mylar) X850 (DAC 001).

Specimen Weight - 1.4217 grams

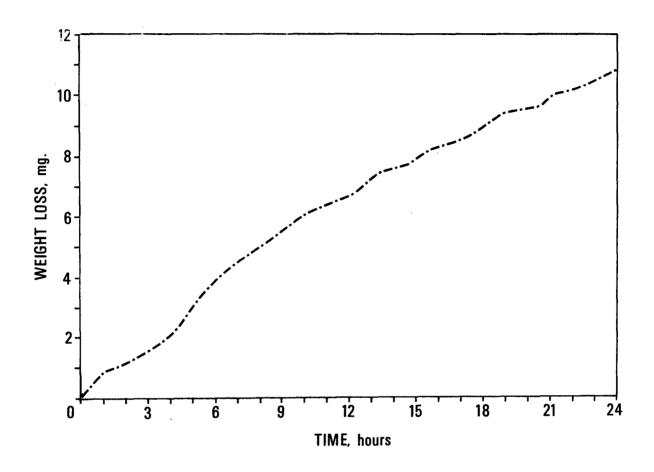


Figure 5. TGA Curve of FM 238 Nitrile/Phenolic (DAC 002).

Specimen Weight - 8.9116 grams

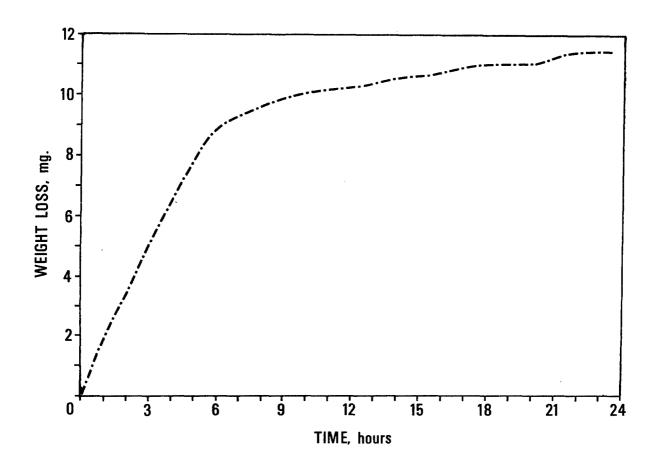


Figure 6. TGA Curve of RTV 731 Silicone (DAC 003). Specimen Weight - 5.3960 grams

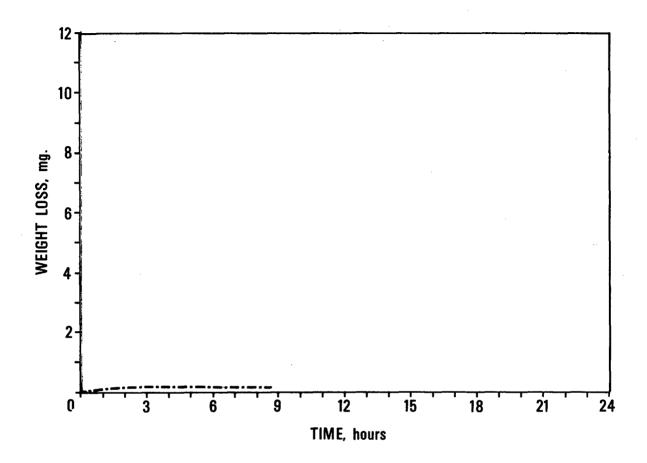


Figure 7. TGA Curve of Saran Wrap Type 18 (DAC 005). Specimen Weight - 2.0140 grams

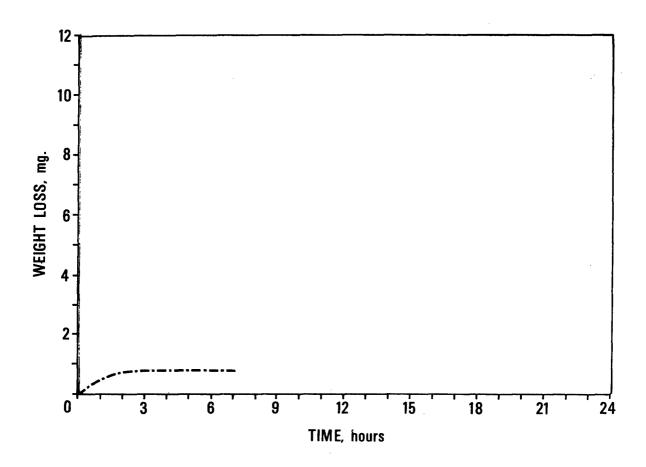


Figure 8. TGA Curve of Aclar Type 33-C (DAC 006).

Specimen Weight - 5.5000 grams

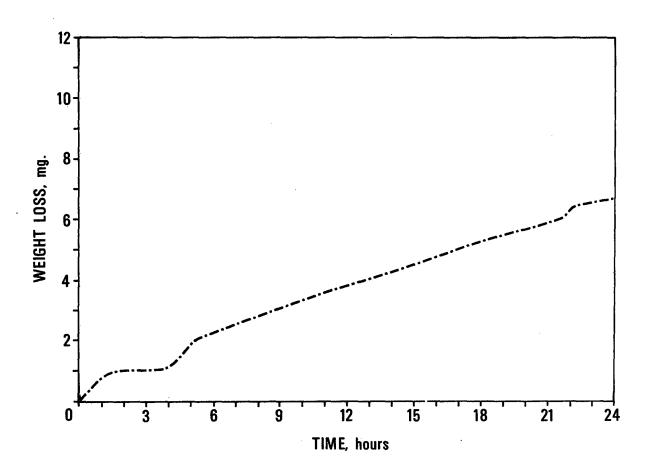


Figure 9. TGA Curve of Shonka A-150 Polyolefin/Polyamide (DAC 007).

Specimen Weight - 10.4906 grams

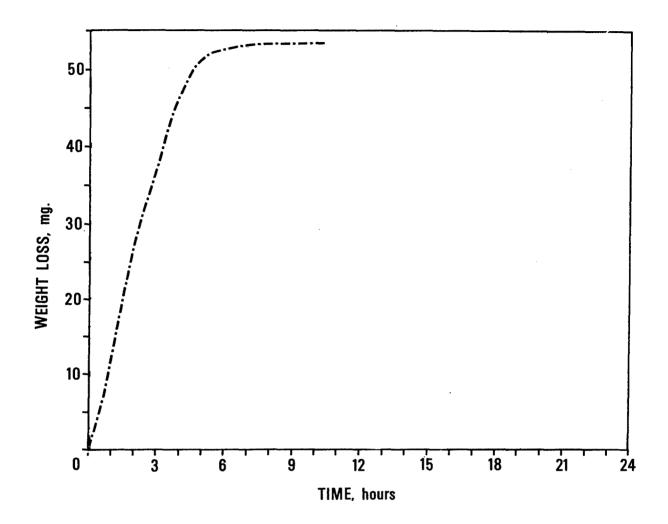


Figure 10. TGA Curve of Spandex Lycra Polyurethane (DAC 008).

Specimen Weight - 1.6140 grams

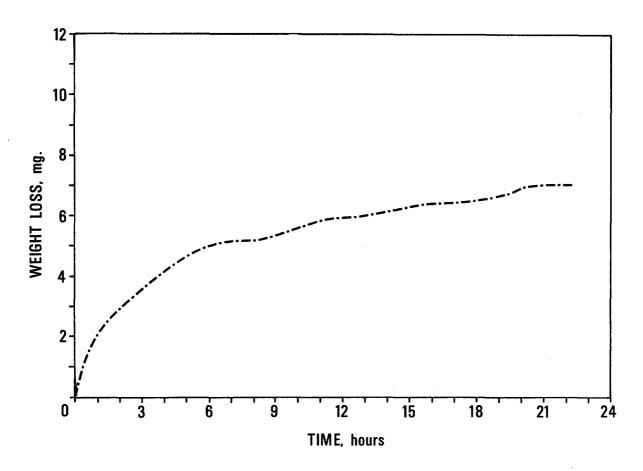


Figure 11. TGA Curve of PR 1535 Polyurethane (DAC 011).

Specimen Weight - 9.3311 grams

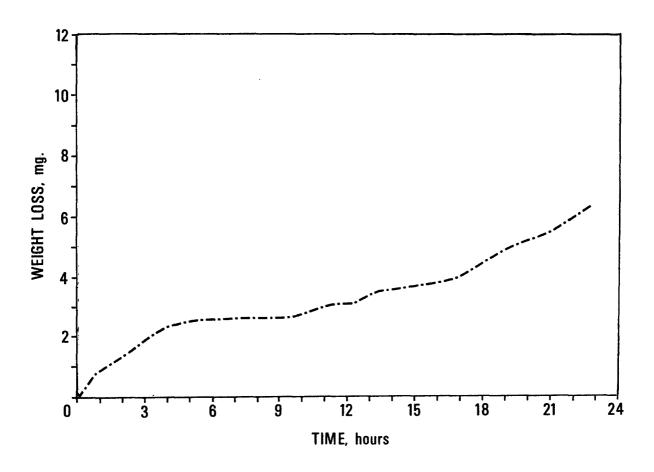


Figure 12. TGA Curve of RTV 615 (DAC 012).

Specimen Weight - 9.9980 grams

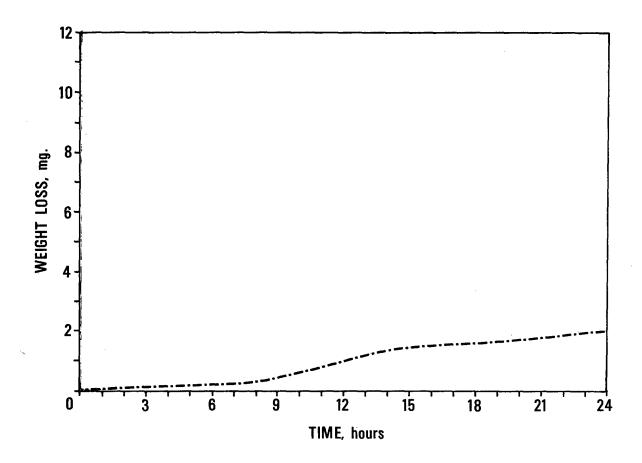


Figure 13. TGA Curve of Stycast 2651/Catalyst II (Epoxy) (DAC 013).

Specimen Weight - 10.0375 grams

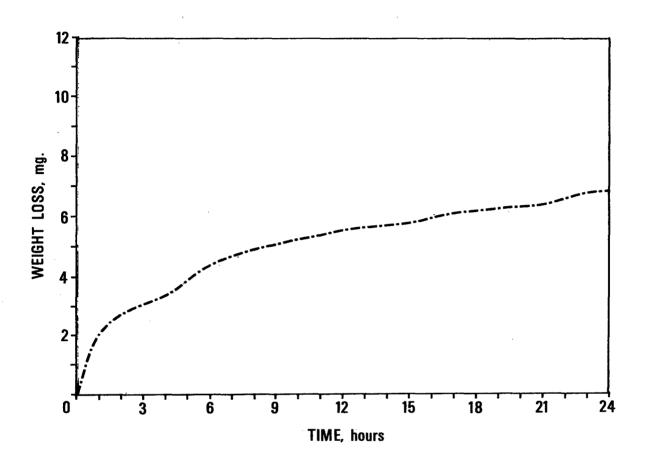


Figure 14. TGA Curve of Hathone HA 7236 (DAC 014).

Specimen Weight - 2.0283 grams

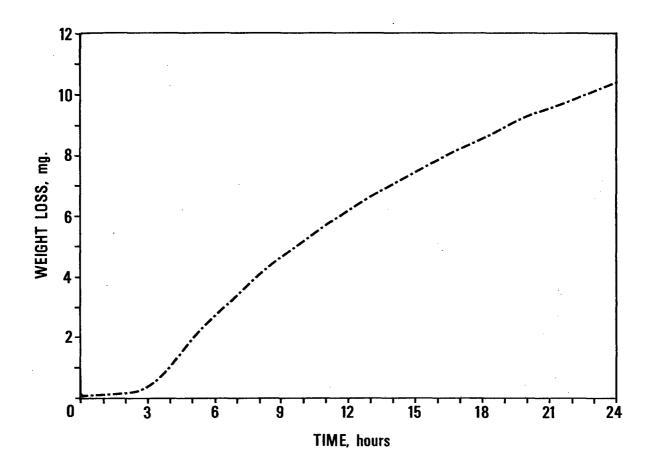


Figure 15. TGA Curve of Butyl Rubber 00996-33L (DAC 017).

Specimen Weight - 10.1100 grams

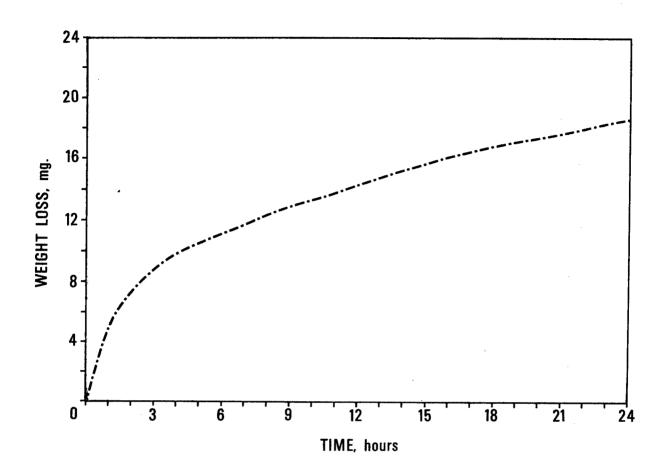


Figure 16. TGA Curve of Polyurethane 00996-39B (DAC 018).

Specimen Weight - 10.0101 grams

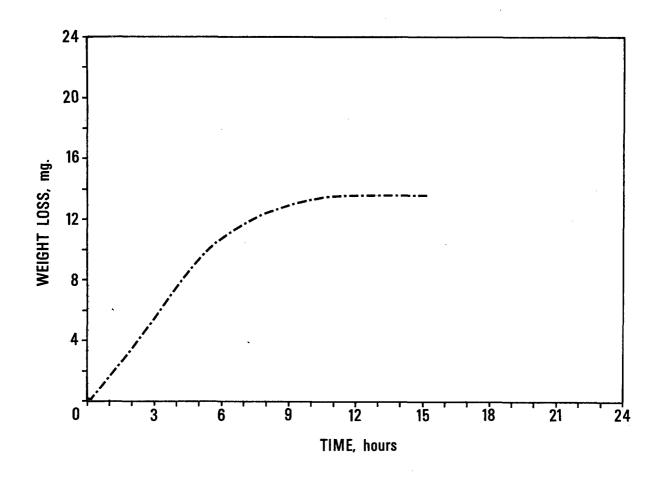


Figure 17. TGA Curve of Silicone 19513-10f (DAC 019).

Specimen Weight - 7.3399 grams

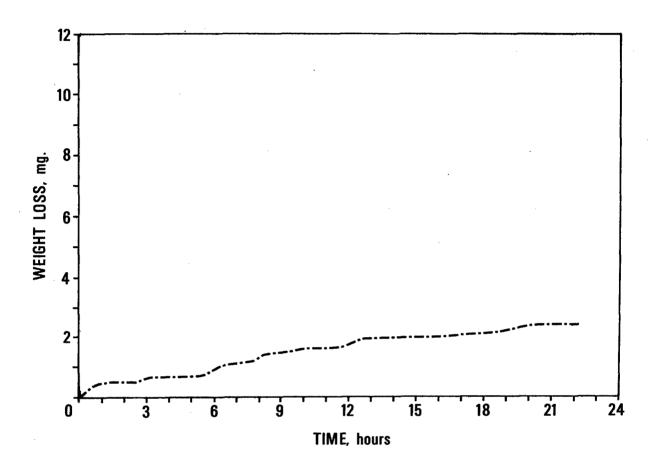


Figure 18. TGA Curve of Boltaron ABS Polymer (DAC 020).

Specimen Weight - 9.3817 grams

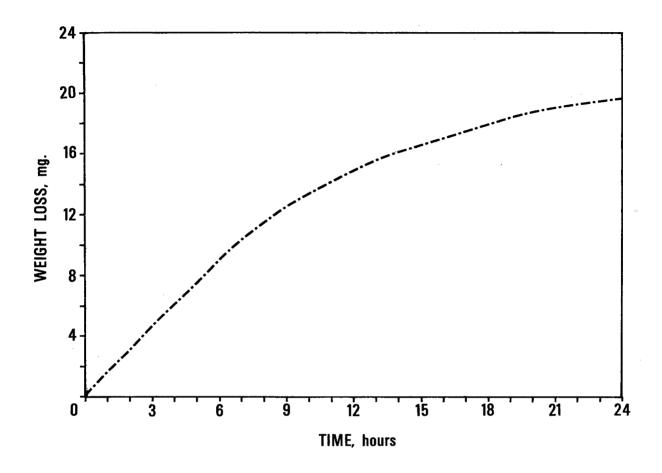


Figure 19. TGA Curve of Epoxy 760A (DAC 021).

Specimen Weight - 9.9900 grams

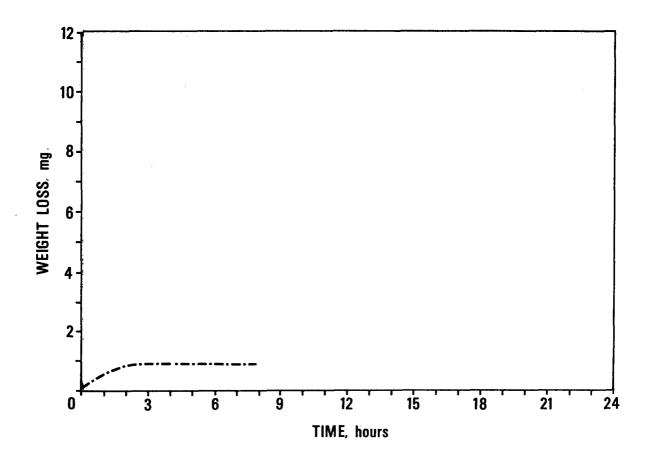


Figure 20. TGA Curve of Merlon 1000 Polycarbonate (DAC 022).

Specimen Weight - 5.0624 grams

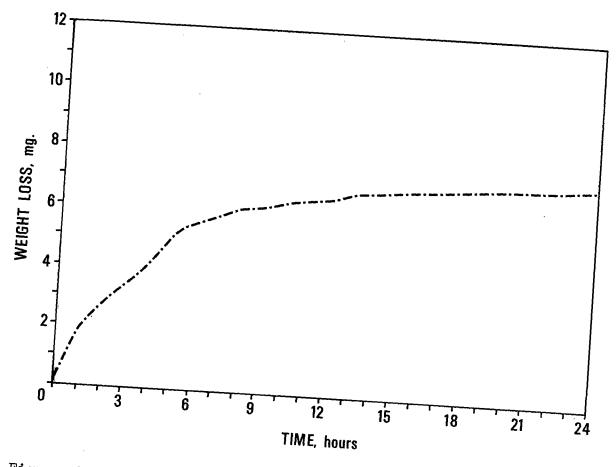


Figure 21. TGA Curve of Silicone, Type A #428/132 (AF 023).

Specimen Weight - 9.8951 grams

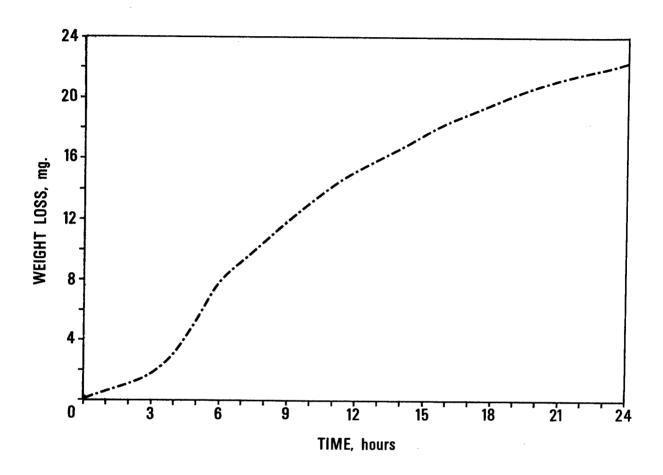


Figure 22. TGA Curve of Bondmaster E611 (Modified Epoxy) (AF 053).

Specimen Weight - 8.5453 grams

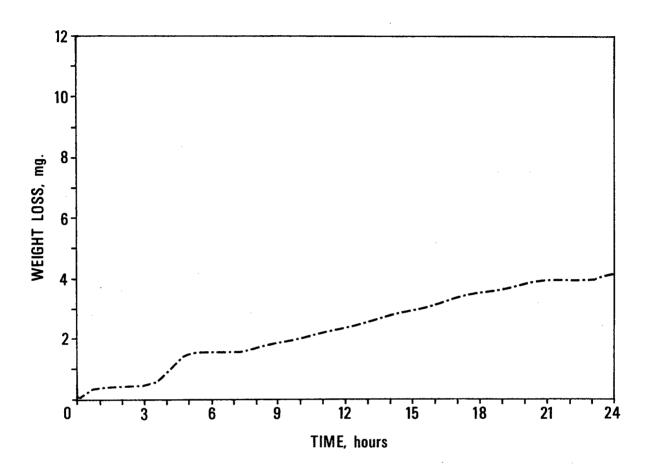


Figure 23. TGA Curve of Epoxy-Polyamide, V-9 Silver Epon 815 (AF 054).

Specimen Weight - 9.9953 grams

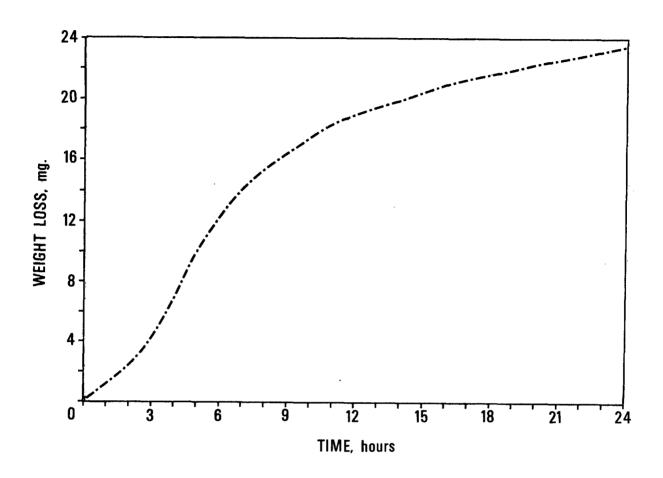


Figure 24. TGA Curve of Silicone EMS 323 (AF 063).

Specimen Weight - 9.3731 grams

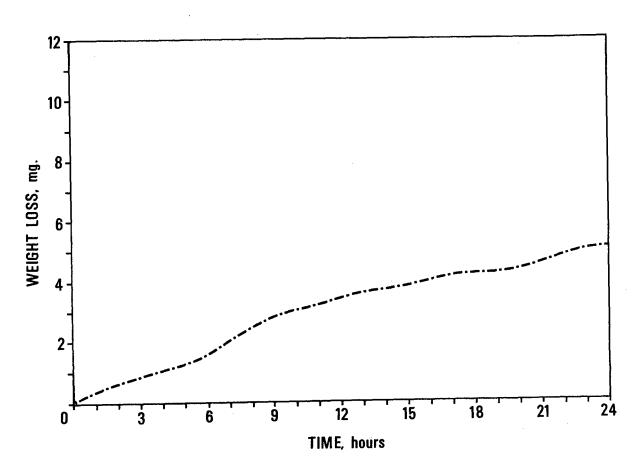


Figure 25. TGA Curve of Viton A EMS 338 (Fluoro Elastomer) (AF 064).

Specimen Weight - 8.8647 grams

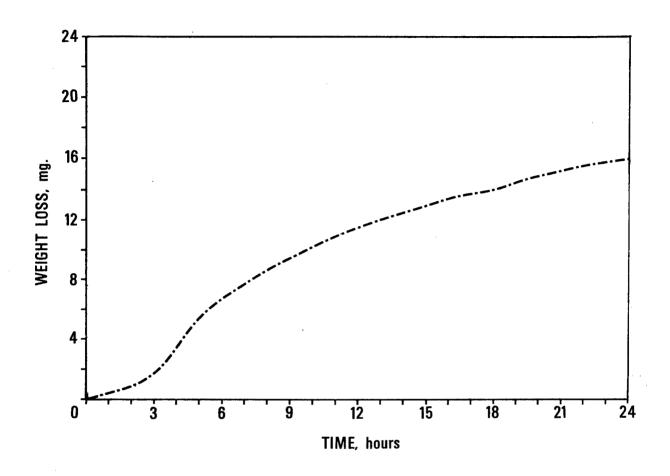


Figure 26. TGA Curve of Silicone Rubber EMS 342 (AF 065).

Specimen Weight - 6.1683 grams

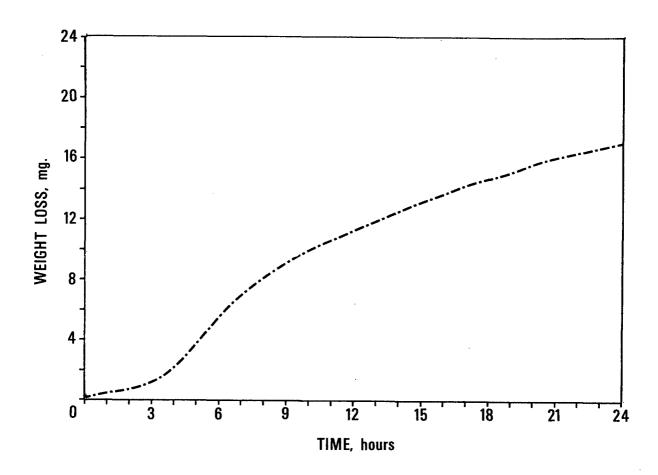


Figure 27. TGA Curve of Silicone Rubber EMS 345 (AF 066).

Specimen Weight - 6.6071 grams

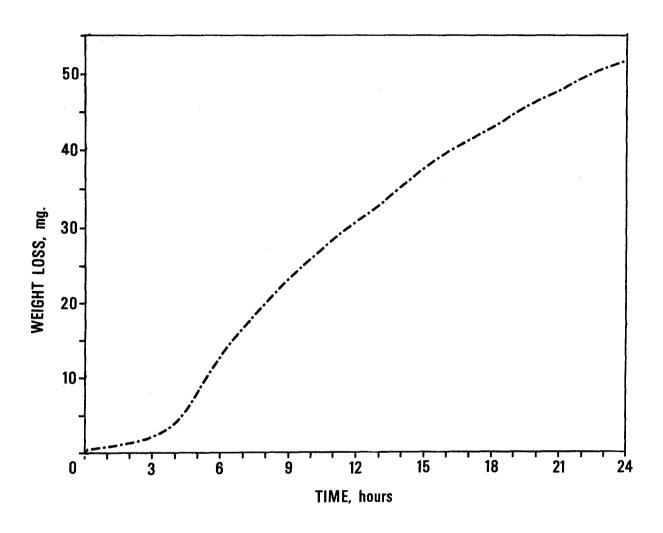


Figure 28. TGA Curve of Hapalon Rubber EMS 355 (AF 068).

Specimen Weight - 10.2164 grams

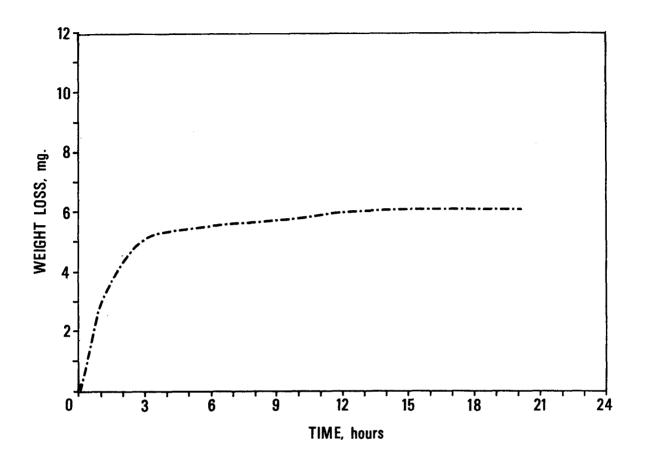


Figure 29. TGA Curve of Urethane EMS 366 (AF 071).

Specimen Weight - 6.0081 grams

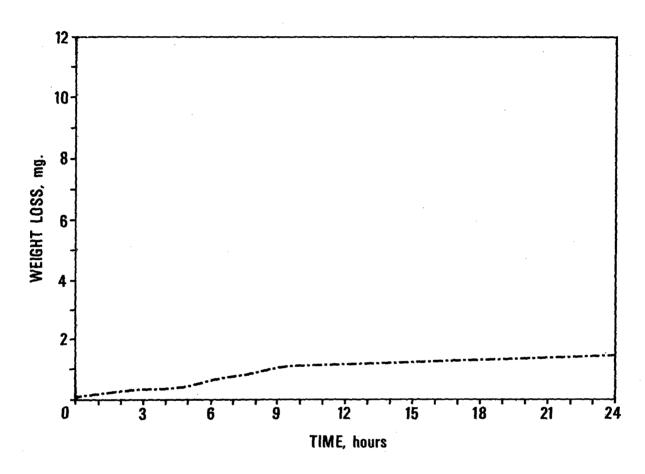


Figure 30. TGA Curve of Polyester (Dacron) Silicone (AF 073). Specimen Weight - 2.8895 grams

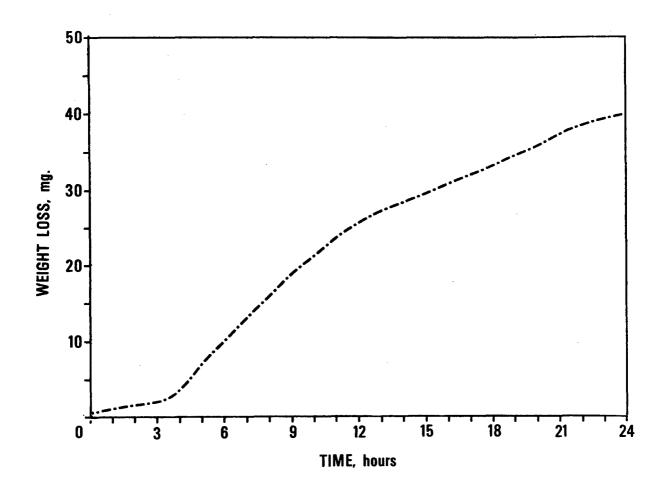


Figure 31. TGA Curve of Epoxy Primer M-602 (AF 203). Specimen Weight - 5.2887 grams

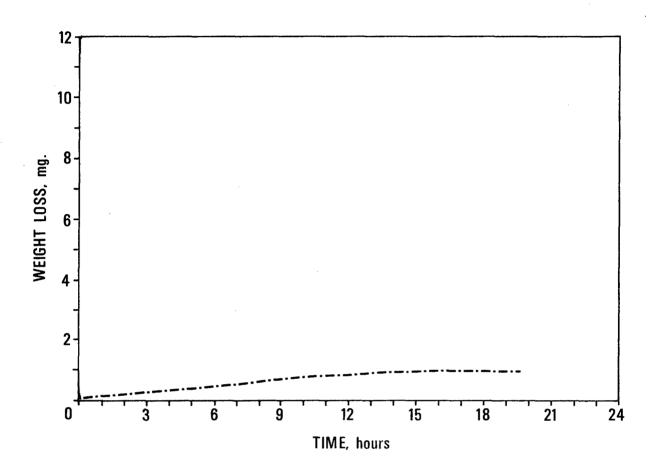


Figure 32. TGA Curve of Irradiated Polyolefin,
Thermofit RNF, CRN (AF 209).

Specimen Weight - 8.1098 g

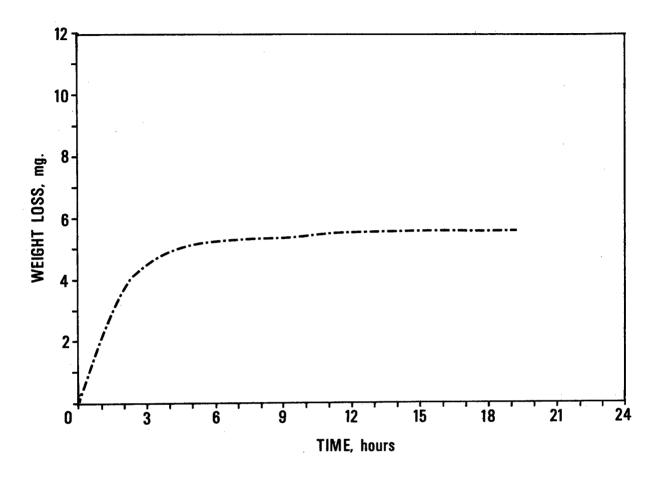


Figure 33. TGA Curve of Mylar Polyester (AF 251).

Specimen Weight - 6.3206 grams

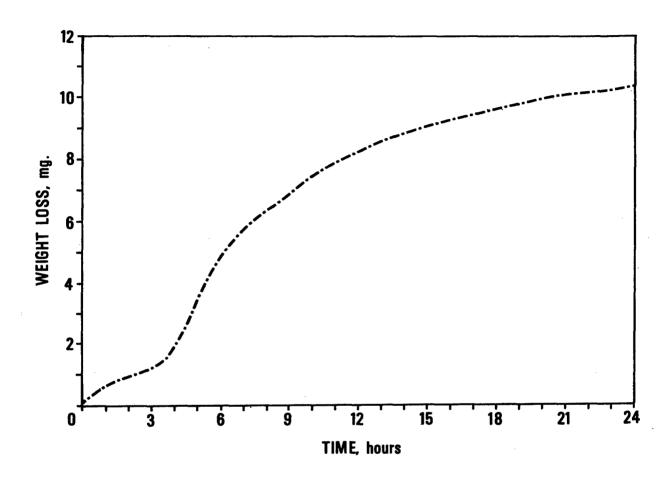


Figure 34. TGA Curve of Irradiated Polyolefin, Thermofit RNF (AF 266).

Specimen Weight - 6.2638 grams

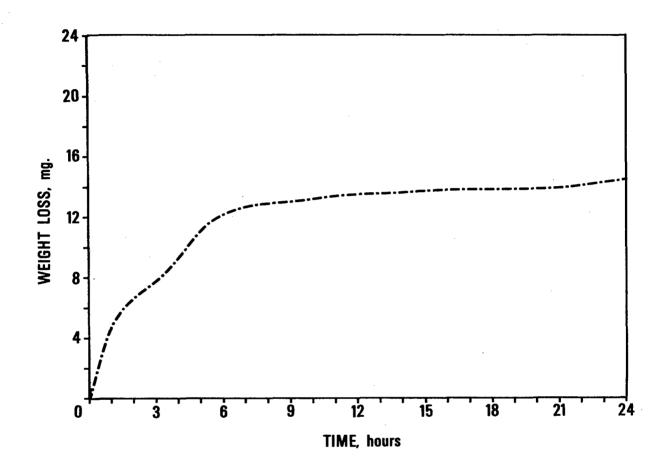


Figure 35. TGA Curve of Eccobond 70C Adhesive (AF 302).

Specimen Weight - 11.1985 grams

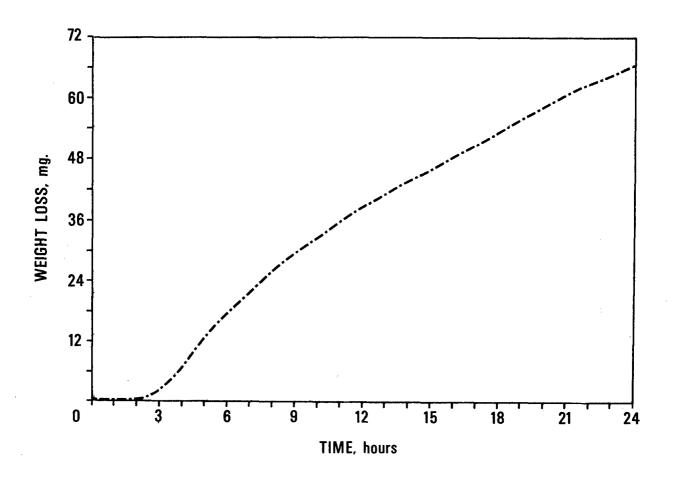


Figure 36. TGA Curve of Thermoplastic Coating, 1169 A/B Coating (AF 325).

Specimen Weight - 2.4288 grams

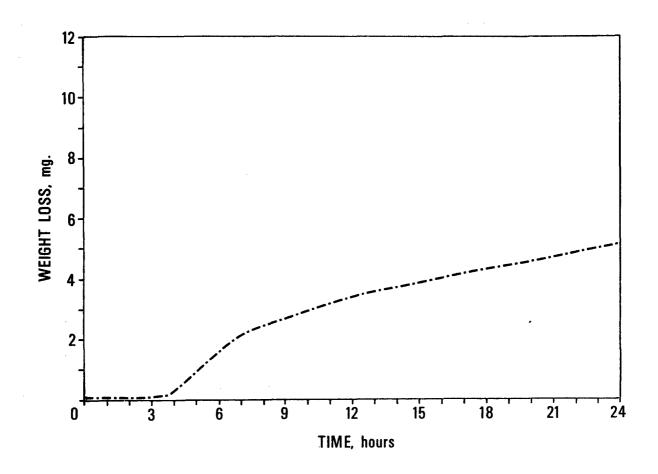


Figure 37. TGA Curve of Stycast 1090 CAT-1 (AF 402). Specimen Weight - 9.5619 grams

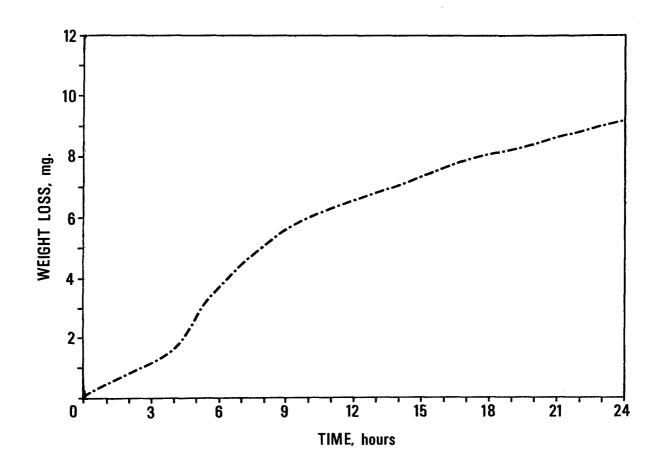


Figure 38. TGA Curve of Stycast 2651-50 CAF1 (AF 403). Specimen Weight - 10.5348 grams

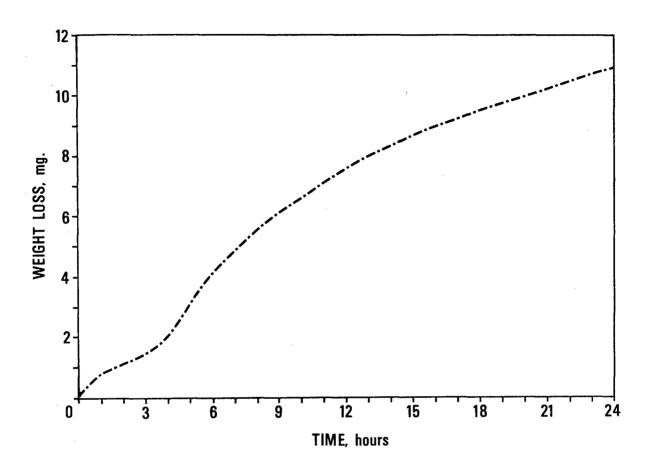


Figure 39. TGA Curve of Silicone RTV 521 (AF 406).

Specimen Weight - 11.7090 grams

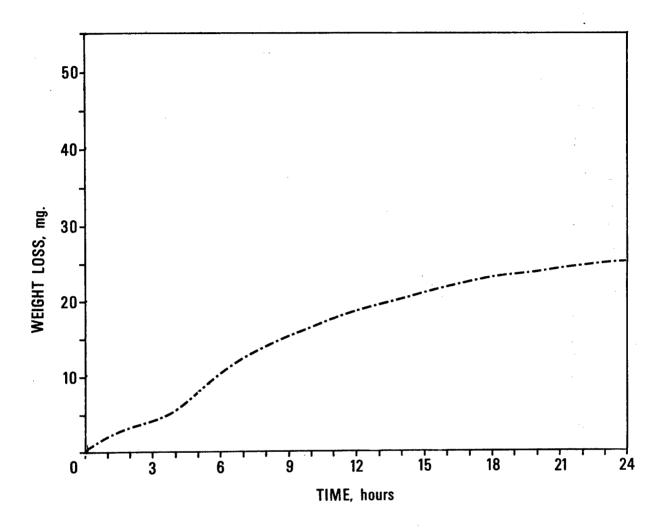


Figure 40. TGA Curve of Adiprene L-100 MCC Adhesive (AF 421).

Specimen Weight - 11.3395 grams

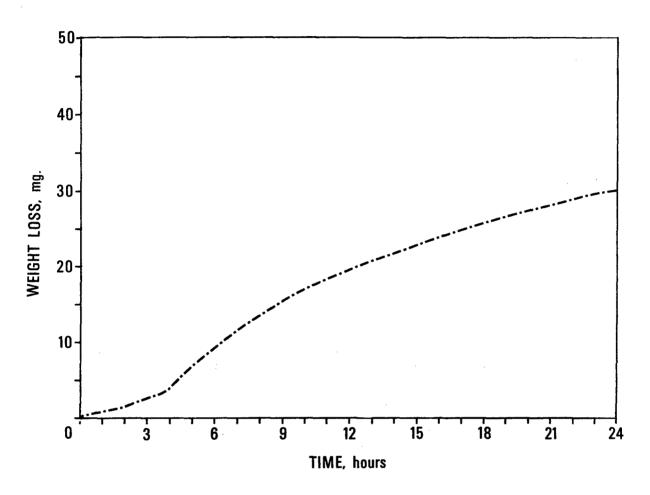


Figure 41. TGA Curve of Silicone RTV 502 Rubber (AF 451).

Specimen Weight - 9.4696 grams

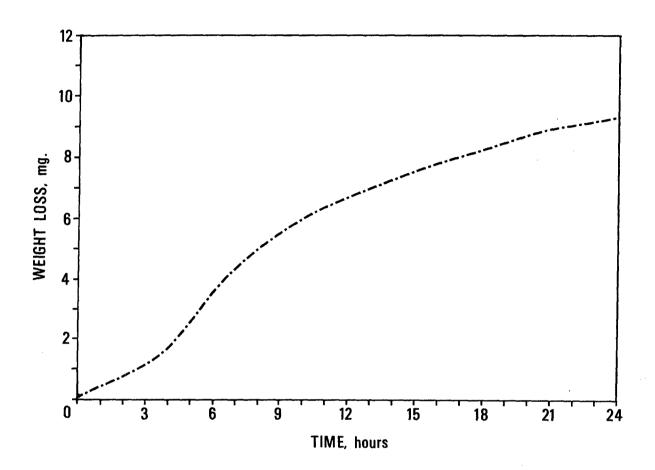


Figure 42. TGA Curve of Silicone Q30079 (AF 454).

Specimen Weight - 6.7228 grams

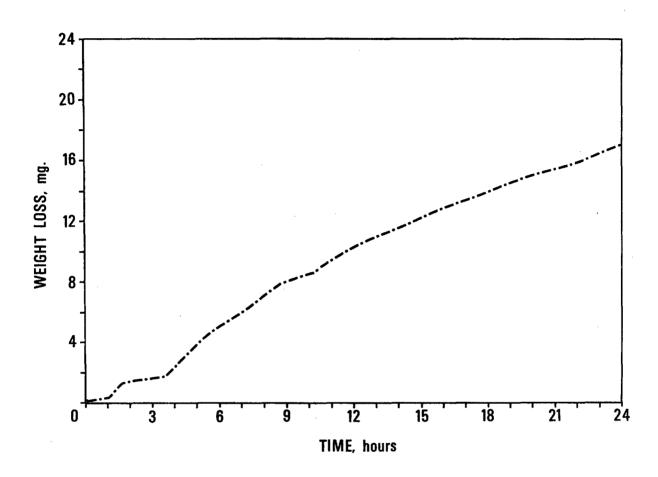


Figure 43. TGA Curve of Silicone RTV 503 (AF 459).

Specimen Weight - 9.9523 grams

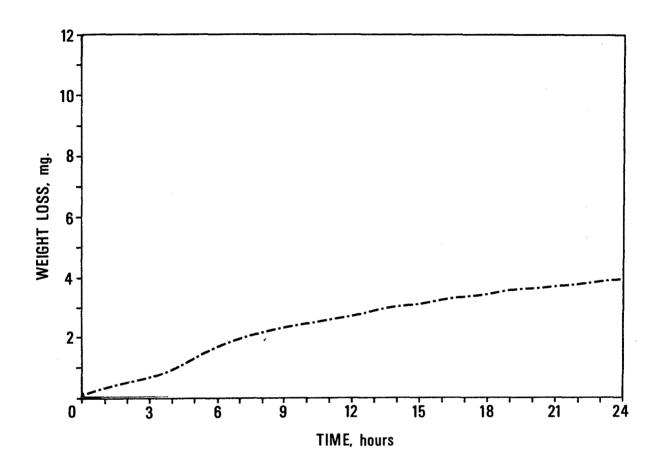


Figure 44. TGA Curve of Tape, Stycast 2651-CAT 9 (AF 495).

Specimen Weight - 10.2746 grams

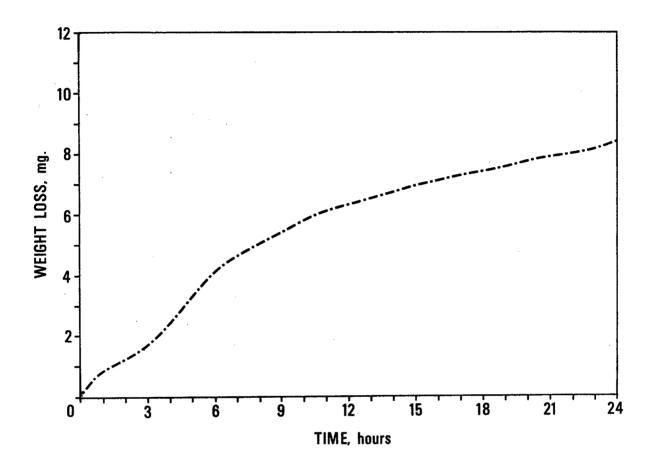


Figure 45. TGA Curve of Oil, Aero Shell #7 (AF 505). Specimen Weight - 7.8232 grams

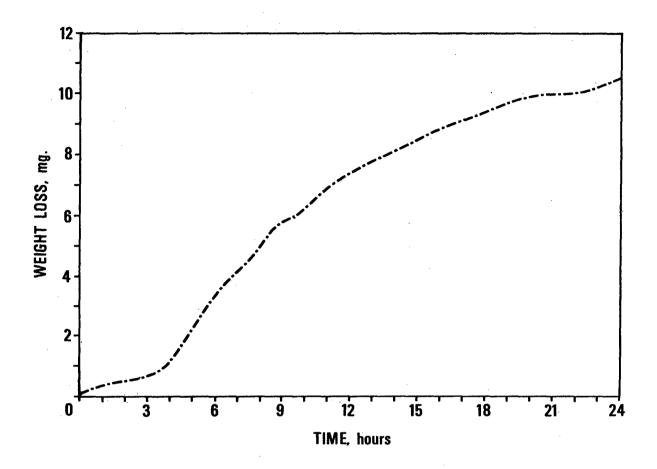


Figure 46. TGA Curve of Paint, Lacquer Stik White (AF 515).

Specimen Weight - 3.2812 grams

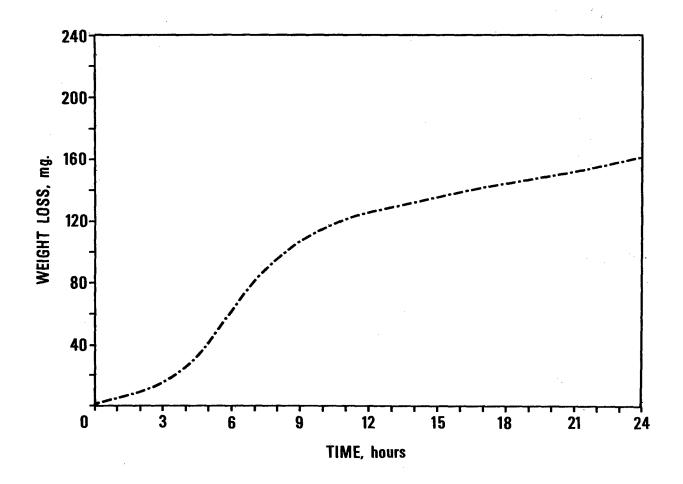


Figure 47. TGA Curve of Conductive Paint (AF 522).

Specimen Weight - 9.8416 grams

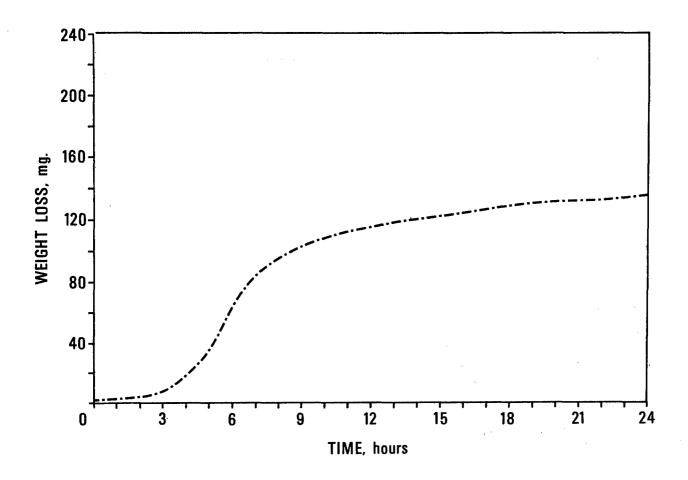


Figure 48. TGA Curve of Resin Emerlon 310 (AF 527). Specimen Weight - 8.9236 grams

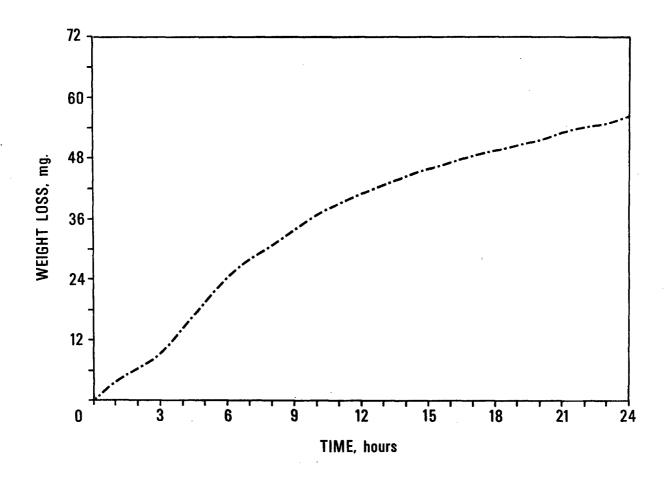


Figure 49. TGA Curve of Varnish E44 (AF 528).

Specimen Weight - 6.5904 grams

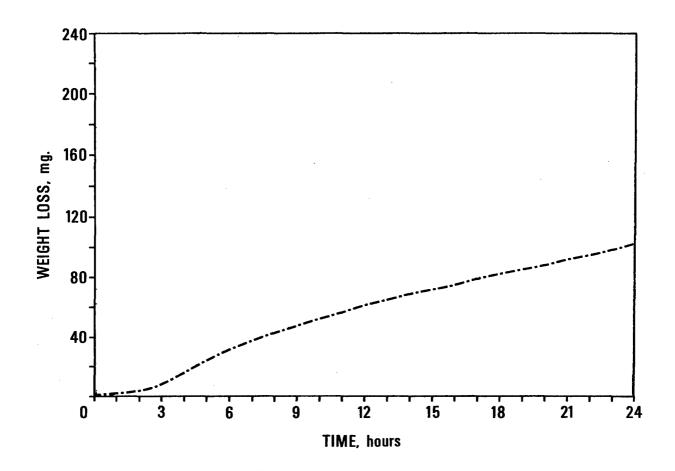


Figure 50. TGA Curve of Polyurethane PC-18 (AF 533). Specimen Weight - 7.3303 grams

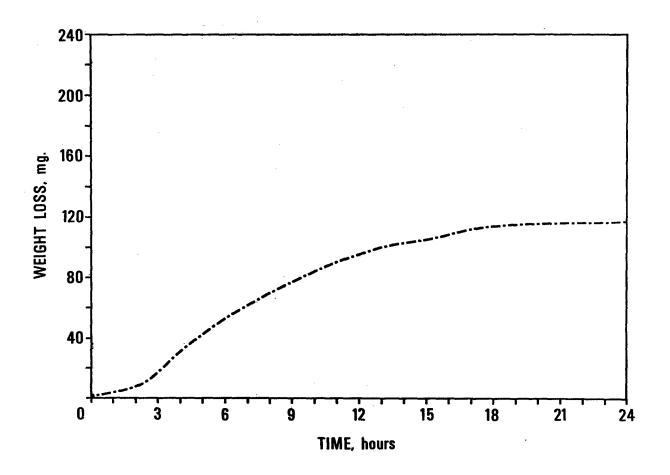


Figure 51. TGA Curve of Ink F-150 (AF 536). Specimen Weight - 3.5743 grams

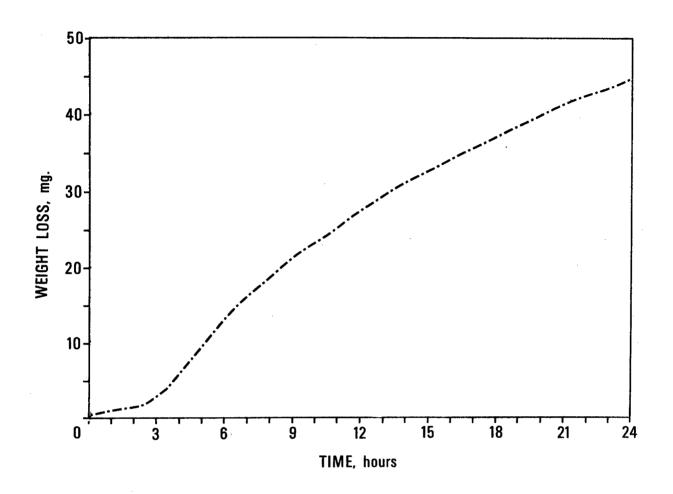


Figure 52. TGA Curve of Ink #41 Black (AF 537).

Specimen Weight - 4.3758 grams

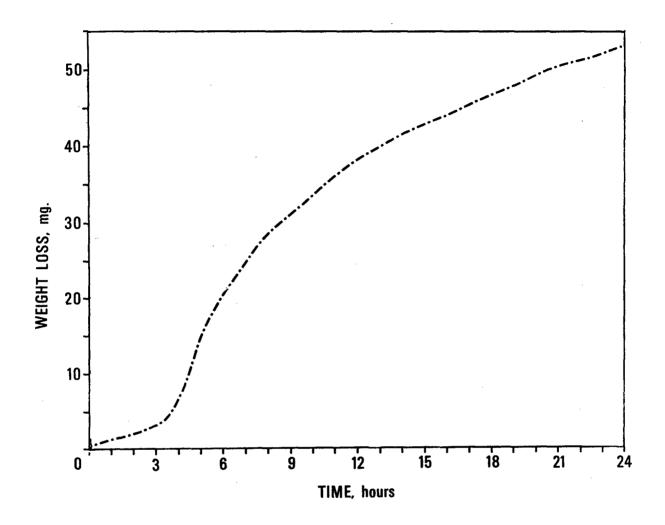


Figure 53. TGA Curve of Ink Red Marking VF-200 (AF 540). Specimen Weight - 2.3888 grams

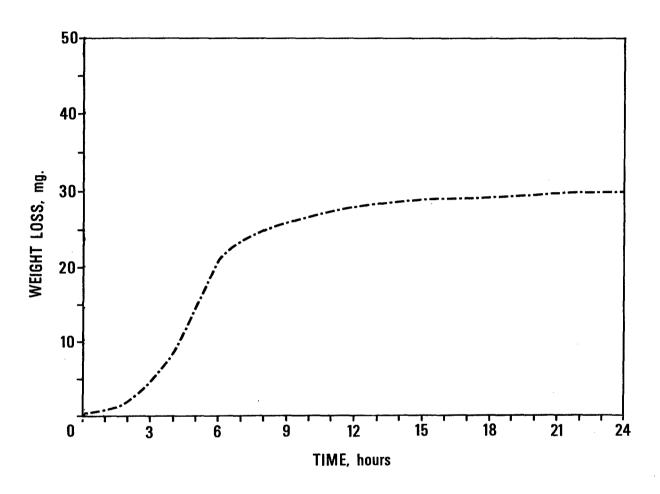


Figure 54. TGA Curve of Nylon Cord Style 18 (AF 551). Specimen Weight - 6.6950 grams

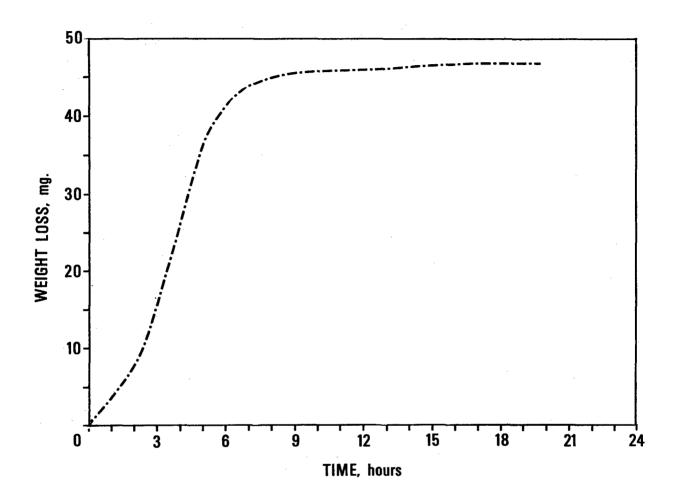


Figure 55. TGA Curve of Nylon Cord Style 21 (AF 552).

Specimen Weight - 8.9585 grams

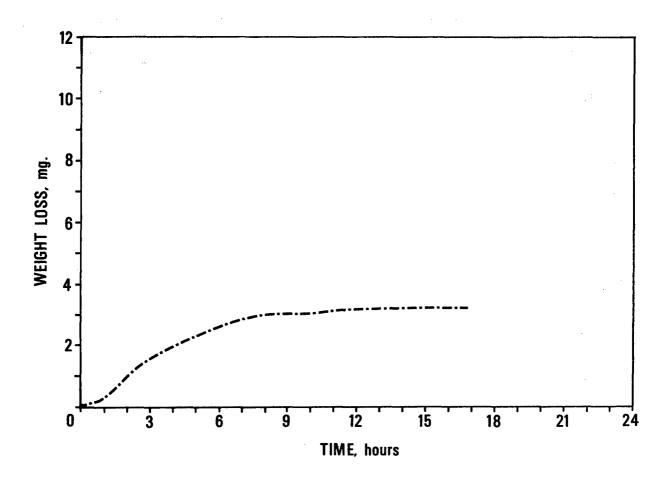


Figure 56. TGA Curve of Lastic 60,000 RF1 (AF 561).

Specimen Weight - 11.2570 grams

APPENDIX II

ANALYTICAL RESULTS
FOR
GAS-OFF EXPERIMENTS

Compounds found as gas-off products from candidate space cabin materials are listed in the following tables. Values for the gas-off product levels are given as: milligrams per 10 grams (mg/10 gms) of the cured candidate material. In some cases, either more or less than 10 g of material was used, but each yield of gas-off products was normalized to that of a 10-g sample.

The order of the tables in this appendix is by Air Force serial number. Names of materials are those submitted by the Air Force.

Table VI

GAS-OFF PRODUCTS FROM NITRILE/PHENOLIC, FM 238

DAC Serial No. 002

Component	(mg/10 g 72 Hours (68°C)	Weight of Component (mg/10 gms Candidate Material) lours 30 Days 60 Days loc) (25°C)	erial) 60 Days (25°C)
Propylene	3.5	2.0	2.0
Propylene Oxide	N.D.	N.D.	0.08
Ethanol	0.3	0.02	N.D.
Jarbon Monoxide	0.005	0.008	0.001
Methane	0.08	0.004	0.004

N.D. = Not Detected

Table VII

GAS-OFF PRODUCTS FROM ACLAR, TYPE 33-C

DAC Serial No. 006

	we (mg/10	<pre>Weight of Component (mg/l0 gms Candidate Material)</pre>	กราชา
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Chlorinated			
Fluorocarbons	2.1	0.08	0.1
Acetone	N D.	ב	0
1 1 1 1 1		• • • • • • • • • • • • • • • • • • • •	400.0
Foliation	N.D.	N.D.	0 0
Toluene	N, D,	, E	
Carbon Monoxide			700°0
	To.o	600.0	0.009
Methane	0.08	0.04	0.05

N.D. = Not Detected

Table VIII

GAS-OFF PRODUCTS FROM POLYOLEFIN/POLYAMIDE, SHONKA A-150

DAC Serial No. 007

	We (mg/lo	Weight of Component (mg/10 gms Candidate Material)	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
C. Unsaturated Hydrocarbon	0.1	N.D.	0.04
Acetone	0.08	N.D.	0.05
Methyl Ethyl Ketone	N.D.	N.D.	0.04
Toluene	N.D.	N.D.	0.001
Diisopropyl Ketone	9.0	0.07	0.3
Di-n-propyl Ketone	0.1	0.008	0.01
Carbon Monoxide	0.03	0.008	0.001
Methane	90.0	0.04	0.006

N.D. = Not Detected

Table IX

GAS-OFF PRODUCTS FROM FOAM, HATHONE HA 7236

DAC Serial No. 014

	mg/log	ignt of Component gms Candidate Mat	; ;er1al)
Component	72 Hours (68°C)	Hours 30 Days 60 Days 8°C) (25°C) (25°C)	60 Days (25°C)
Freon-11	4.5	2.7	3.1
Trichloroethylene	0.02	N.D.	N. N.
Xylene	0.01	N.D.	N.D.
Carbon Monoxide	0.01	0.004	0.001
Methane	<0.01	0.04	900.0

N.D. = Not Detected

Table X

GAS-OFF PRODUCTS FROM BUTYL RUBBER, 00996-33L

DAC Serial No. 017

	We:	Weight of Component (mg/10 gms Candidate Material)	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Isobutylene Diisobutylene	0.3	0.08	7.0
Triisobutylene	0.03	0.01	0.3
Tetraisobutylene	0.03	N.D.	N.D.
Toluene	N.D.	N.D.	ħ0.0
Xylene	N.D.	0.002	0.001
Carbon Monoxide	0.05	0.02	0.001
Methane	0.07	0.04	900.0

N.D. = Not Detected

Table XI

GAS-OFF PRODUCTS FROM POLYURETHANE, 00996-39B

DAC Serial No. 018

	Weight of mg/10 gms Ca (68°C) N.D. N.D. 0.06 0.2 0.15
Weight of Component ours 30 Days 60 Days oc) (25°C) (25°C) o.08 0.03 n.D. 0.01 o.2 0.01 o.1 0.05 o.01 0.05 o.04 0.04	

N.D. = Not Detected.

Table XII

GAS-OFF PRODUCTS FROM SILICONE, 19513-10f

DAC Serial No. 019

	We (mg/10	Weight of Component (mg/10 gms Candidate material)	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	N.D.	0.02	0.01
Ethanol	0.04	0.1	90.0
Toluene	N.D.	90.0	0.004
Butanol	N.D.	N.D.	0.01
Acetophenone	0.04	N.D.	N.D.
2-Phenyl-2-propanol	0.03	N.D.	N.D.
Carbon Monoxide	0.01	0.02	0.008
Methane	0.08	0.05	ħ0°0

N.D. - Not Detected

Table XIII

GAS-OFF PRODUCTS FROM ABS POLYMER, BOLTARON

DAC Serial No. 020

	Web	Weight of Component (mg/10 gms Candidate Material)	(נסיים)
Component	72 Hours (68°C)	30 Days (25°C)	(25°C)
Ethanol	N.D.	N.D.	0.01
n-Propanol	N.D.	N.D.	0.01
Trichloroethylene	0.02	N.D.	0.02
Toluene	0.008	N.D.	0.03
Xylene	0.02	N.D.	0.02
Styrene	0.2	0.04	0.11
Methylstyrene	0.02	N.D.	0.01
Carbon Monoxide	900.0	0.004	0.001
Methane	0.05	0.04	900.0

N.D. = None Detected

Table XIV

GAS-OFF PRODUCTS FROM EPOXY, 760 A

DAC Serial No. 021

	We	Weight of Component	([(† % (
Component	(mg/10 72 Hours (68°C)	gms candidate mad 30 Days (25°C)	60 Days (25°C)
2-Butanone	1.8	0.5	0.09
Carbon Monoxide	0.03	0.1	0.008
Methane	0.08	0.02	0.04

Table XV

GAS-OFF PRODUCTS FROM SILICONE, TYPE A #428/132

AF Serial No. 023

	We:	Weight of Component (mg/10 gms Candidate Material)	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.02	N.D.	0.01
Ethanol	0.01	N D.	0.002
Benzene	0.02	N.D.	N.D.
2-Methyl-4-pentanone	0.21	0.03	0.03
Toluene	0.17	60.0	0.09
n-Butanol	0.17	0.02	0.02
Xylenes	0.22	0.09	0.09
Carbon Monoxide	10.0	900.0	0.004
Methane	0.04	0.05	0.05

N.D. = Not Detected

Table XVI

GAS-OFF PRODUCTS FROM EPOXY, BONDMASTER E611

AF Serial No. 053

i di	me)	Weight of Component (mg/10 gms Candidate Material)	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Ethylene	2.8	0.04	0.08
Acetone	0.02	N.D.	N.D.
Ethanol	0.01	N.D.	N.D.
C4-C6 Hydrocarbons	0.10	N.D.	N.D.
Carbon Monoxide	0.05	0.003	900.0
Methane	0.08	0.04	0.05

N.D. = Not Detected

Table XVII

GAS-OFF PRODUCTS FROM EPOXY/POLYAMIDE, V-9 SILVER EPON 815

AF Serial No. 054

	wej mg/10	<pre>Welght of Component (mg/l0 gms Candidate Material)</pre>	คหาคา)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	600.0	N.D.	N.D.
Ethanol	600.0	N.D.	N.D.
2-Butanol	0.1	N.D.	0.001
Toluene	0.002	N°D.	N.D.
n-Butanol	0.1	N.D.	N.D.
Xylenes	0.005	N.D.	N.D.
Carbon Monoxide	90.0	0.005	900.0
Methane	0.05	40.0	0.05

N.D. = Not Detected

Table XVIII

GAS-OFF PRODUCTS FROM BLACK SILICONE, EMS 323

AF Serial No. 063

N.D. N.D. 0.006 0.04 N.D. N.D. N.D. N.D. Weight of Component(mg/10 gms Candidate Material)ours30 Days0C)(25°C) 0.005 N.D. 0.05 N.D. N.D. N.D. N.D. N.D. 72 Hours (68°C) 0.007 0.05 0.03 0.01 0.07 0.01 0.01 0.01 0.05 Carbon Monoxide Component C4 Unsaturated Hydrocarbons Silicone Oil Benzene Acetone Toluene Xylenes Ethanol Methane

N.D. = Not Detected

Table XIX

GAS-OFF PRODUCTS FROM SILICONE, 342 RUBBER

AF Serial No. 065

	We	Weight of Component	
	72 Hours	(mg/lo gms candidate Material) urs 30 Days 60	erial) 60 Days
Component	(0,89)	(52°C)	(25°C)
Methanol	0.03	0.01	0.02
Silicone Oil	0.30	0.10	0.12
Toluene	¿00°0	0°002	0.01
Xylene	0.021	0.01	0.02
Carbon Monoxide	900.0	0.001	0.003
Methane	60.0	0.1	0.05

Table XX

GAS-OFF PRODUCTS FROM SILICONE, EMS 345 RUBBER

AF Serial No. 066

	Weigh (mg/lo gms	Weight of Component (mg/10 gms Candidate Material)	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.007	N.D.	N.D.
Methanol	0.15	0.008	0.02
Silicone Oil	1.5	90.0	0.12
t-Butanol	0.050	0.003	0.007
Toluene	0.007	<0.001	0.001
Carbon Monoxide	0.007	0.007	0.003
Methane	0.1	20.0	0.05

N.D. = Not Detected

Table XXI

GAS-OFF PRODUCTS FROM RUBBER, EMS 355

AF Serial No. 068

	We	ight of Component	
	(mg/l0	(mg/10 gms Candidate Material)	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	600.0	0.002	0.005
Toluene	0.013	0.003	0.005
Xylene	0.021	0.002	0.005
Carbon Monoxide	0.007	0.003	0.003
Methane	0.04	0.04	0.04

Table XXII

GAS-OFF PRODUCTS FROM EMS 366 URETHANE

AF Serial No. 071

	Welg Welg	Welght of Component	(• • • • • • • • • • • • • • • • • • •
Component	72 Hours (68°C)	("E/IOURS CAMULUACE MACELIAL) (25 Hours 30 Days 6((68°C) (25°C)	60 Days (25°C)
Acetone	0.009	N.D.	<0.001
Methanol	0.018	N.D.	<0.001
2-Propanol	0.010	N.D.	<0.001
Carbon Monoxide	0.02	0.002	0.004
Methane	60.0	0.04	0.04

N.D. = Not Detected

Table XXIII

GAS-OFF PRODUCTS FROM SILICONE, DACRON

AF Serial No. 073

Component Acetone Silicone Oil Ethanol Toluene Carbon Monoxide	(mg/10 g 72 Hours (68°C) 0.03 0.03 0.06 0.009	(mg/10 gms Candidate Material) urs 30 Days 60 C) (25°C) (2 3 N.D. N 6 0.01 0 0 0.001 0 1 0.008 0.008 0	erial) 60 Days (25°C) N.D. N.D. 0.04 0.003
Methane	0.1	0.2	0.2

N.D. = Not Detected

Table XXIV

GAS-OFF PRODUCTS FROM EPOXY, PRIMER M-602

AF Serial No. 203

	weig (mg/10 gr	<pre>weight of Component (mg/l0 gms Candidate Material)</pre>	rial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	5.0	0.02	0.03
C4-C6 Hydrocarbons	η.Ο	N.D.	N.D.
2-Butanone	81.0	2.0	5.0
n-Propanol	0.0	0.04	0.1
2-Methyl-1-propanol	7.0	0.1	0.2
Toluene	1.0	η.0	7.0
n-Butanol	92.0	6.1	10.0
Xylenes	120.0	0.3	0.7
Carbon Monoxide	0.03	0.01	0.02
Methane	90.0	0.02	0.03

N.D. = Not Detected

Table XXV

GAS-OFF PRODUCTS FROM TUBING, THERMOFIT RNF, CRN

AF Serial No. 209

60 Days (25°C) 0.005 0.002 N.D. N.D. N.D. N.D. N.D. (mg/10 gms Candidate Material Weight of Component 30 Days (25°C) 0.003 0.04 N.D. N.D. N.D. N.D. N.D. N.D. 72 Hours (68°C) 0.009 0.014 900.0 0.007 0.002 0.002 0.007 0.04 Ce Hydrocarbon(s) Carbon Monoxide Component Acetaldehyde n-Butanol Acetone Toluene Methane Ethanol

N.D. = Not Detected

Table XXVI

GAS-OFF PRODUCTS FROM POLYESTER, MYLAR FILM

AF Serial No. 251

	Wei	Weight of Component (mg/10 gms Candidate Material)	, to
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Chloroform	0.10	0.01	0.02
Acetone	0.12	N.D.	0.004
Benzene	0.004	N.D.	<0.00
Toluene	0.001	N.D.	<0.001
Carbon Monoxide	0.002	0.002	0.002
Methane	0.04	0.04	0.04

N.D. - Not Detected

Table XXVII

GAS-OFF PRODUCTS FROM TUBING, THERMOFIT RNF

AF Serial No. 266

N.D. = Not Detected

Table XXVIII

GAS-OFF PRODUCTS FROM ADHESIVE, ECCOBOND 70C

AF Serial No. 302

	Weight (mg/10 gms	Weight of Component (mg/10 gms Candidate Material)	al)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Formaldehyde	0.004	N.D.	N.D.
Acetaldehyde	0.008	N.D.	N.D.
Methyl Formate	0.004	N.D.	N.D.
Ethanol	0.04	<0.001	0.001
Benzene	0.002	N.D.	N.D.
Formic Acid	0.002	N.D.	N.D.
Toluene	0.002	N.D.	N.D.
Carbon Monoxide	0.1	0.005	0.005
Methane	90.0	0.04	0.04

N.D. = Not Detected

Table XXIX

GAS-OFF PRODUCTS FROM STYCAST, 1090 CAT-1

AF Serial No. 402

N.D. = Not Detected

Table XXX

GAS-OFF PRODUCTS FROM STYCAST, 2651-50 CAFI

AF Serial No. 403

	Weight Weight	Weight of Component (mg/10 gms Candidate Material)	rial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.012	N.D.	<0.001
Benzene	0.005	N.D.	N.D.
Carbon Monoxide	0.01	0.004	0.002
Methane	0.04	0.05	0.04

N.D. = Not Detected

Table XXXI

GAS-OFF PRODUCTS FROM SILICONE, RTV 521

AF Serial No. 406

erial)	60 Days (25°C)	N.	0.01	0.03	22.0	N D	0.03
Weight of Component (mg/10 gms Candidate Material)	30 Days (25°C)	N.D.	0.01	0.02	0.81	0.003	0.05
Wei (mg/10 g	(68°C)	0.01	†0°0	0.18	1.4	0.002	0.04
	Component	Methanol	Acetone	Ethanol Isopropanol	Silicone Oil	Carbon Monoxide	Methane

N.D. = Not Detected

Table XXXII

GAS-OFF PRODUCTS FROM ADHESIVE, ADIPRENE L-100 MCC

AF Serial No. 421

	We	Weight of Component	
	(mg/10	(mg/10 gms Candidate Material)	erial)
Component	(5°89)	(25°C)	(25°C)
Methanol	90.0	0.018	0.022
Ethanol	0.018	0.009	0.012
Benzene	0.002	N.D.	N.D.
Toluene	0.001	<0.00	<0.001
Xylene	0.003	N.D.	0.001
Carbon Monoxide	0.002	0.001	0.001
Methane	ħ0°0	0.04	0.03

N.D. = Not Detected

Table XXXIII

GAS-OFF PRODUCTS FROM SILICONE, RTV 502 RUBBER

AF Serial No. 451

	We (mg/mg/	Weight of Component	F 1
Component	72 Hours (68°C)	urs 30 Days 60 (25°C)	60 Days (25°C)
Acetone	0.04	N.D.	N.D.
Silicone Oil	2.1	0.05	0.15
n-Propanol	3.9	1.2	7.6
n-Butanol	0.03	N.D.	N.D.
Ethylbenzene	0.03	N. D.	. n. n.
Xylenes	0.03	N.D.	N.D.
Carbon Monoxide	0.02	0.002	0.004
Methane	0.05	0.05	0.05

N.D. = Not Detected

Table XXXIV

GAS-OFF PRODUCTS FROM SILICONE FOAM, Q30079

AF Serial No. 454

	Wei (mg/lo	Weight of Component (mg/l0 gms Candidate Material)	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.02	0.001	0.001
Ethanol	600.0	N.D.	N.D.
Silicone Oil	0.15	600.0	0.02
Propanol	900.0	N.D.	N.D.
Toluene	0.035	600.0	0.01
Butanol	0.02	0.002	0.004
Xylenes	0.09	0.008	0.010
Carbon Monoxide	0.02	0.002	0.002
Methane	0.05	0.05	0.05

N.D. = Not Detected

Table XXXV

GAS-OFF PRODUCTS FROM SILICONE FOAM, RTV 503

AF Serial No. 459

	NO.	Weight of Component	
	(mg/10	(mg/10 gms Candidate Material)	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.02	N.D.	N.D.
Ethanol	5.0	2.8	4.9
Silicone Oil	2.2	N.D.	N.D.
Xylenes	0.04	N.D.	N.D.
Carbon Monoxide	0.02	0.001	0.002
Methane	0.05	0.04	0.05

N.D. = Not Detected

Table XXXVI

GAS-OFF PRODUCTS FROM TAPE, STYCAST 2651-CAT 9

AF Serial No. 495

	we] (mg/10	weight of Component (mg/10 gms Candidate Material)	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.004	0.003	0.007
Ethanol	0.04	<0.001	0.01
Toluene	90.0	N.D.	0.001
n-Butanol	900.0	N.D.	N.D.
Xylenes	0.003	N.D.	N.D.
Carbon Monoxide	0.02	0.004	0.003
Methane	0.05	0.05	0.05

N.D. = Not Detected

Table XXXVII

GAS-OFF PRODUCTS FROM OIL, AERO SHELL #7

AF Serial No. 505

	Wei (mg/lo	Weight of Component (mg/10 gms Candidate Material)	ค.ร.
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.007	N.D.	N.D.
Ethanol	0.02	N.D.	N.D.
n-Propanol	0.02	N.D.	N.D.
2-Butanol	0.01	N.D.	N.D.
Toluene	0.02	0.001	0.002
n-Butanol	0.02	N.D.	N.D.
Xylene	0.01	N.D.	N.D.
Naphthalene	0.02	N.D.	N.D.
Carbon Monoxide	0.008	0.005	0.008
Methane	0.05	0.05	0.07

N.D. = Not Detected

Table XXXVIII

GAS-OFF PRODUCTS FROM PAINT, LACQUER STIK WHITE

AF Serial No. 515

	(mg/1	f Compon ndidate	ent Material)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
rmaldehyde	2.3	N.D.	N.D.
C4 Unsaturated Hydrocarbon(s)} Acetaldehyde	0.32	0.28	0.39
Cs Unsaturated Hydrocarbon(s)	•	•	0.21
Fropionaldenyde Ffr:1fcmm:te	7.7	0 (ب د
Acetone		60.0	0.0
C ₆ Unsaturated Hydrocarbon(s)	∞	0	
Butyraldehyde	w.	2	ς.
2-Butanone	4	0	0
C, Unsaturated Hydrocarbon(s)	•	0.43	ω.
Valeraldehyde	•	÷	N
	•	÷	↑.
Cs Unsaturated Hydrocarbon(s)	0.79	60.0	0.11
Z-Bucen-1-01 Hexanone	· C	, ה	
Cyclopentanone	• •	z z	A A
3-Methyl-1-butanol	0.26	N.D.	
Co Unsaturated Hydrocarbon(s)	0	N.D.	N.D.
Cyclohexanol	ď		N.D.
3-Methylcyclohexanol	۲.	•	N.D.
Acetic Acid		N.D.	N.D.
Carbon Monoxide	25.2	9.8	14.5

N.D. = Not Detected

Table XXXIX

GAS-OFF PRODUCTS FROM PAINT, CONDUCTIVE

AF Serial No. 522

		Weight of Component O gms Candidate Material	erial)
Component	(68°C)	30 Days (25°C)	60 Days (25°C)
C3-C4 Hydrocarbons	0.03	N.D.	N.D.
Acetone	2.2	4.0	1.1
Ethanol	3.2	6.0	7.5
Propanol	0.08	0.02	0.7
Propylacetate	1.3	0.3	1.2
Toluene	0.3	0.07	0.2
2-Butanol	6.0	2.0	2.7
l-Butanol	5.2	1.2	. ω.
Xylene	0.04	N.D.	N.D.
2-Ethoxyethylacetate	0.09	0.02	0.09
2-n-Propoxyethanol	0.3	40.0	0.1
2-n-Butoxyethanol	2.8	0.2	1.2
2-n-Butoxyethylacetate	0.04	N.D.	N.D.
Carbon Monoxide	1.1	0.2	0.03
Methane	0.03	90.0	0.07

N.D. = Not Detected

Table XL

GAS-OFF PRODUCTS FROM RESIN, EMERLON 310

AF Serial No. 527

	Weight	Weight of Component (mg/10 gms Candidate Material	ท ำ ลา)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Air			
Acetone	4.0	0.04	0.1
Ethanol	21	1.8	6.9
Propylacetate	0.8	0.1	9.0
Toluene	9.0	0.08	0.3
2-Butanol	2.3	0.3	1.2
1-Butanol	56	2.5	8.0
Butylacetate	7.2	0.8	3.3
Xylene	7.0	90.0	7.0
2-Ethoxyethanol	1.6	0.08	0.3
2-Ethoxyethylacetate	9.2	٦°6	4.1
Carbon Monoxide	0.02	0.01	0.005
Methane	0.05	0.05	20.0
Phenol	3.3	N.D.	N.D.

N.D. = Not Detected

Table XLI

GAS-OFF PRODUCTS FROM VARNISH, E44

AF Serial No. 528

	Wei	Weight of Component (mg/10 gms Candidate Material)	ค. เกา
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.21	0.02	0.01
Ethanol	2.2	01.0	0.30
2-Butanone	0.20	0.01	0.007
Benzene	0.05	0.003	0.002
Methylisobutylketone	11.7	18.4	18.0
Toluene	25.2	3.4	3.5
Butanol	8.9	0.02	0.02
Xylenes	5.7	0.36	0.38
C3-C4 Alkylbenzenes	0.58	N.D.	N.D.
Carbon Monoxide	0:05	0.007	0.003
Methane	0.05	0.05	90.0

N.D. = Not Detected

Table XLII

GAS-OFF PRODUCTS FROM COATING, POLYURETHANE PC-18

AF Serial No. 533

	Wej (mg/l0 g	Weight of Component (mg/10 gms Candidate Material)	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.61	0.005	0.007
Ethanol	0.08	N.D.	0.005
Benzene	0.02	N.D.	0.001
2-Butanol	0.72	N.D.	N.D.
Toluene	0.22	0.004	0.008
n-Butanol	0.84	N.D.	N.D.
Xylenes	11.8	1.4	2.7
2-Ethoxyethylacetate	6.9	0.32	0.68
C ₃ -Alkylbenzenes	ħ.O	N.D.	N.D.
Carbon Monoxide	0.008	0.005	0.001
Methane	0.04	90.0	0.08

N.D. = Not Detected

Table XLIII

GAS-OFF PRODUCTS FROM CORD, STYLE 18 NYLON

AF Serial No. 551

	We.	Weight of Component	F
Component	72 Hours (68°C)	urs 30 Days 60 (25°C)	erial) 60 Days (25°C)
Acetone	0.5	0.005	0.016
Toluene	0.009	N.D.	0.003
n-Butanol	0.03	N.D.	0.009
1,3-Dioxane	0.03	N.D.	N.D.
1-Methylmorpholine	0.03	N.D.	N.D.
Jarbon Monoxide	0.03	900.0	0.002
Methane	90.0	0.05	90.0

N.D. = Not Detected

Table XLIV

GAS-OFF PRODUCTS FROM CORD, STYLE 21 NYLON

AF Serial No. 552

	We	Weight of Component	
Component	(mg/10 72 Hours (68°C)	(mg/10 gms Candidate Material) urs 30 Days 60	(25°C)
Acetone	0.02	100.0>	<0.001
Ethanol	600.0	<0.001	<0.001
sec-Butanol	0.01	<0.001	0.002
Toluene	0.04	0.002	0.004
n-Butanol	0.11	0.003	900.0
Xylene	0.01	N.D.	N.D.
Carbon Monoxide	0.01	200.0	0.002
Methane	0.05	0.05	90.0

N.D. = Not Detected

APPENDIX III

REPRESENTATIVE GAS CHROMATOGRAMS
FOR
GAS-OFF EXPERIMENTS

The gas chromatograms shown in this appendix were obtained on an F&M Scientific Corporation Model 810 Research Gas Chromatograph. Instrument conditions and column specifications are listed in Table XLV. Since retention times tended to shift somewhat due to column aging, a standard mixture was used as a day-to-day reference.

The gas chromatograms are representative of a particular candidate material. Comparison of peak intensities in chromatograms for different candidate materials should be made with care, since sensitivity factors and quantities of atmosphere taken for analysis vary.

Chromatograms appear in order of their Air Force serial numbers. Names of materials are those submitted by the Air Force.

Table XLV

GAS CHROMATOGRAPHIC INSTRUMENT CONDITIONS

All samples were analyzed using a flame ionization detector and a F&M Model 810 Research Gas Chromatograph.

Instrument Conditions

I. Column: 12-ft x 1/8-in O.D. Stainless Steel, 7% neopentyl-glycolsuccinate on 60/80 mesh Gas-Pack F + 20-ft x 1/4-in. O.D., 5% Carbowax 20M on 60/80 mesh Gas-Pack F.

Column Temperature: programmed 50°-185°C @ 8°C/min.

Detector Temperature: 275°C

Injection Port Temperature: 250°C

Flow Split: 1:9

Flow Rate: 60 ml/min.

Range: 10

Attenuation: X8, or as noted

Sample Size: 25 cc of gas

II. Column: 20-ft x 1/4-in O.D. Stainless Steel, 20% Triton X-305 on 60/80 mesh Gas Chrom Z.

Column Temperature: programmed 50°-170°C @ 8°C/min.

Detector Temperature: 300°C

Injection Port Temperature: 250°C

Flow Split: none

Flow Rate: 60 ml/min.

Range: 10

Attenuation: X8, or as noted

Sample Size: 50 cc of gas

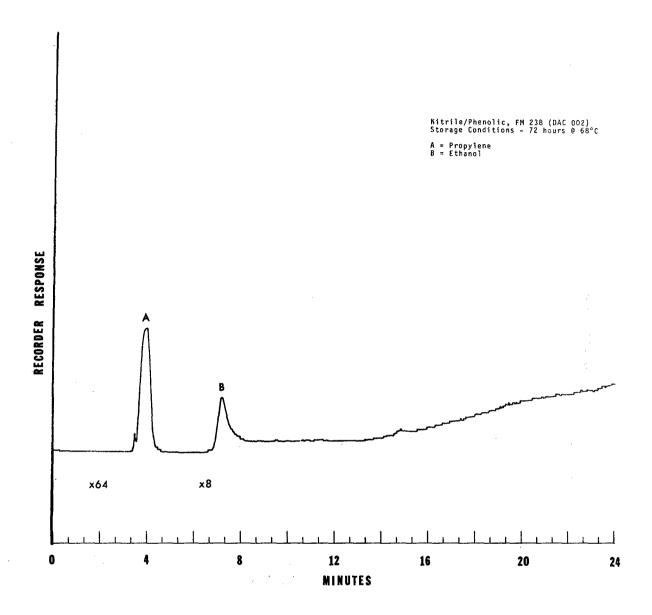


Figure 57. Gas Chromatogram of Gas-Off Products from Nitrile/Phenolic, FM 238 (DAC 002) (72 hours @ 68°C).

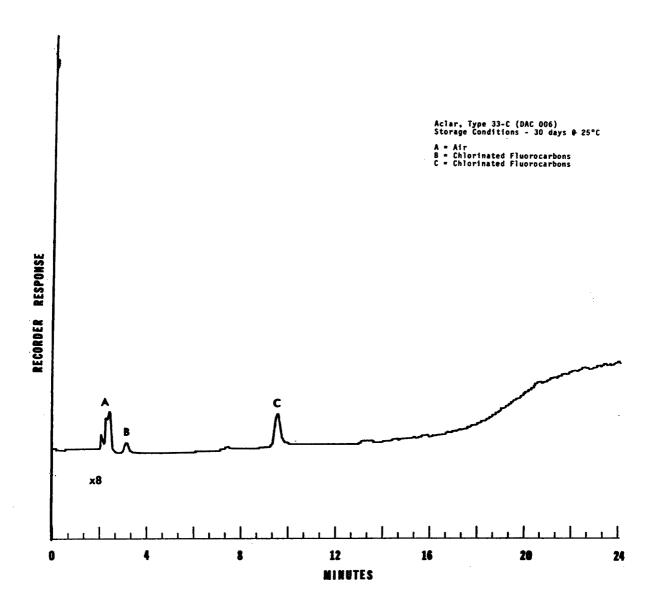


Figure 58. Gas Chromatogram of Gas-Off Products from Aclar, Type 33-C (DAC 006) (30 days @ 25°C).

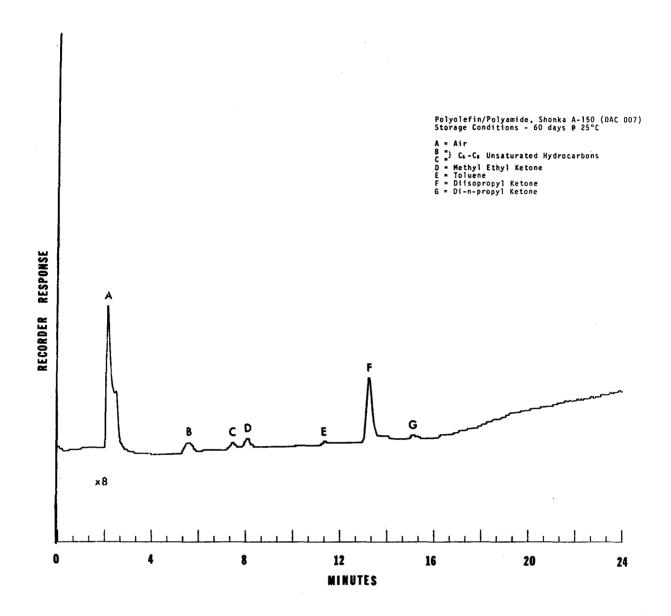


Figure 59. Gas Chromatogram of Gas-Off Products from Polyolefin/Polyamide, Shonka A-150 (DAC 007) (60 days @ 25°C).

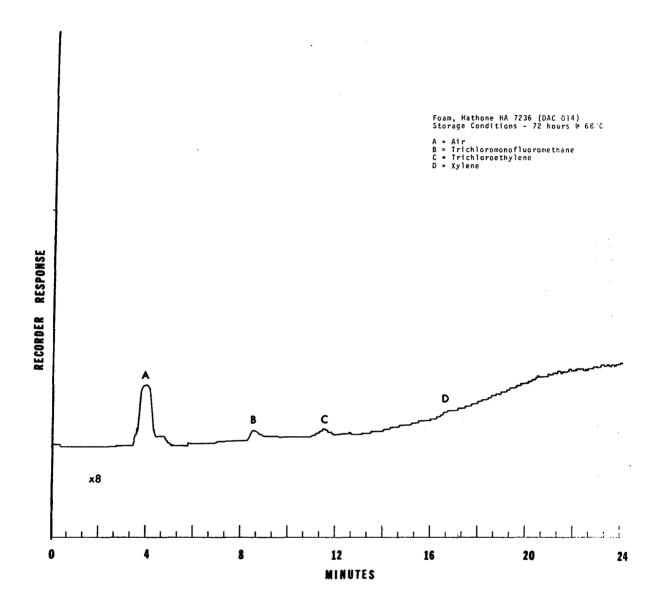


Figure 60. Gas Chromatogram of Gas-Off Products from Foam, Hathone HA 7236 (DAC 014) (72 hours @ 68°C).

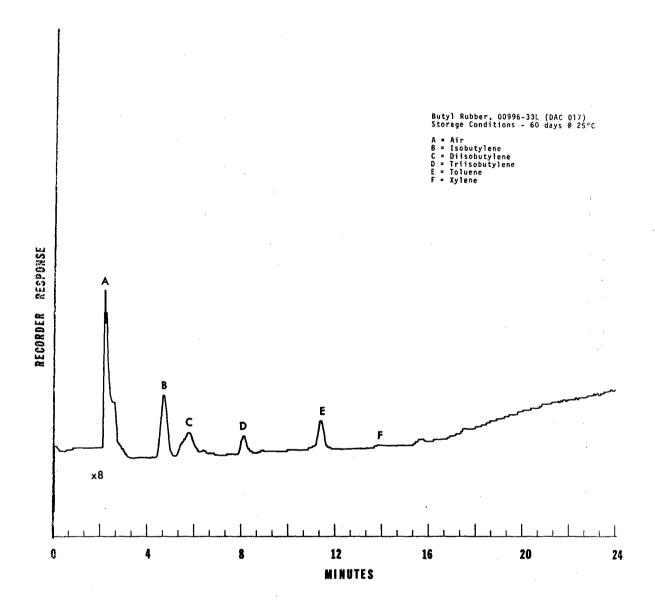


Figure 61. Gas Chromatogram of Gas-Off Products from Butyl Rubber, 00996-33L (DAC 017) (60 days @ 25°C).

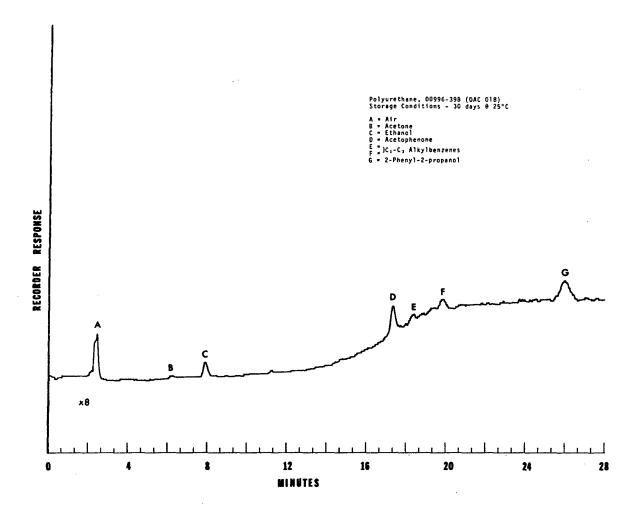


Figure 62. Gas Chromatogram of Gas-Off Products from Polyurethane, 00996-39B (DAC 018) (30 days @ 25°C).

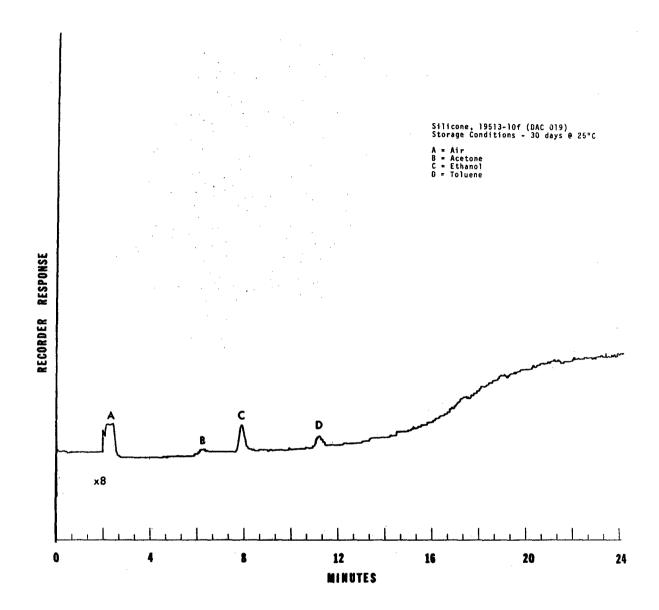


Figure 63. Gas Chromatogram of Gas-Off Products from Silicone, 19513-10f (DAC 019) (30 days @ 25°C).

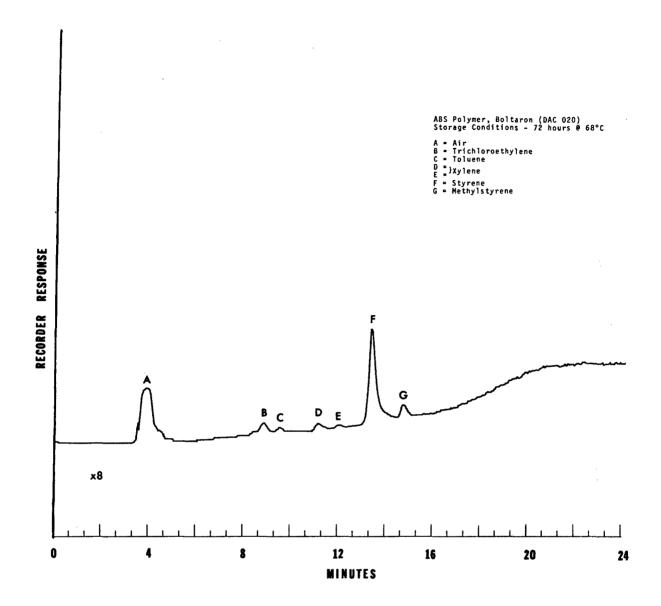


Figure 64. Gas Chromatogram of Gas-Off Products from ABS Polymer, Boltaron (DAC 020) (72 hours @ 68°C).

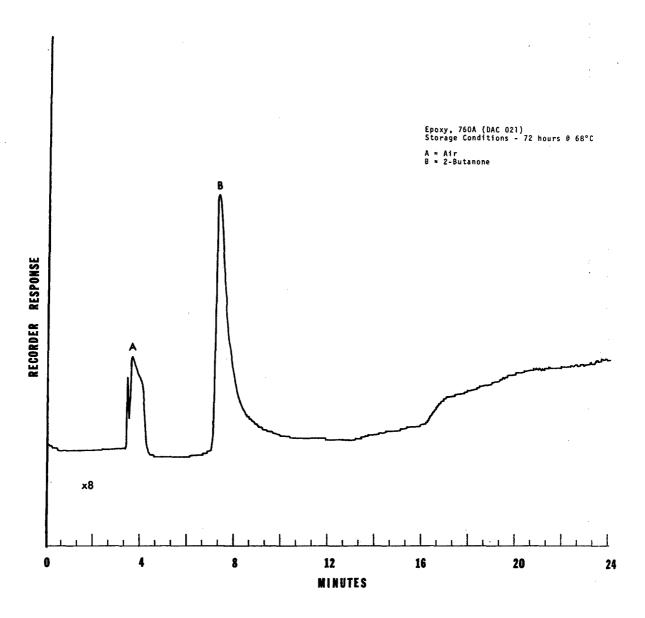


Figure 65. Gas Chromatogram of Gas-Off Products from Epoxy, 760A (DAC 021) (72 hours @ 68°C).

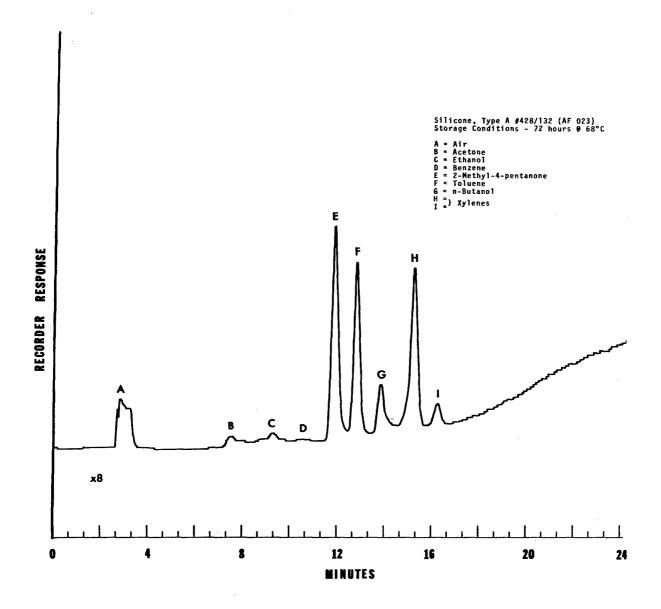


Figure 66. Gas Chromatogram of Gas-Off Products from Silicone, Type A #428/132 (AF 023) (72 hours @ 68°C).

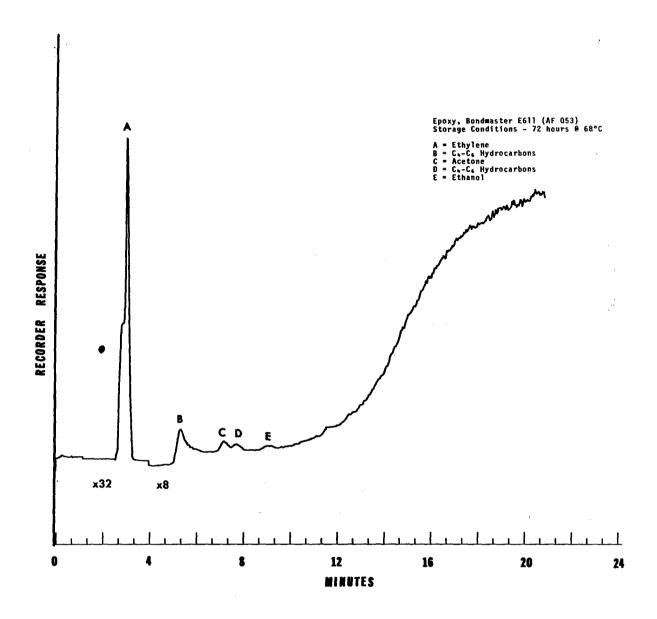


Figure 67. Gas Chromatogram of Gas-Off Products from Epoxy, Bondmaster E611 (AF 053) (72 hours @ 68°C).

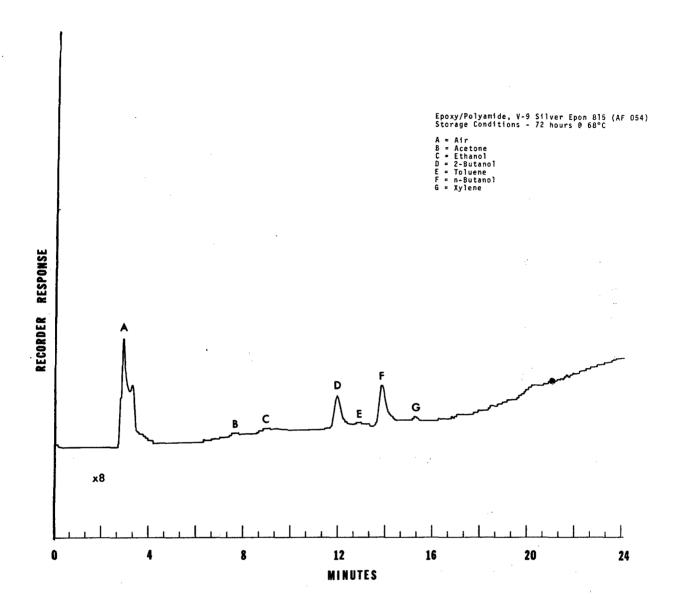


Figure 68. Gas Chromatogram of Gas-Off Products from Epoxy/Polyamide, V-9 Silver Epon 815 (AF 054) (72 hours @ 68°C).

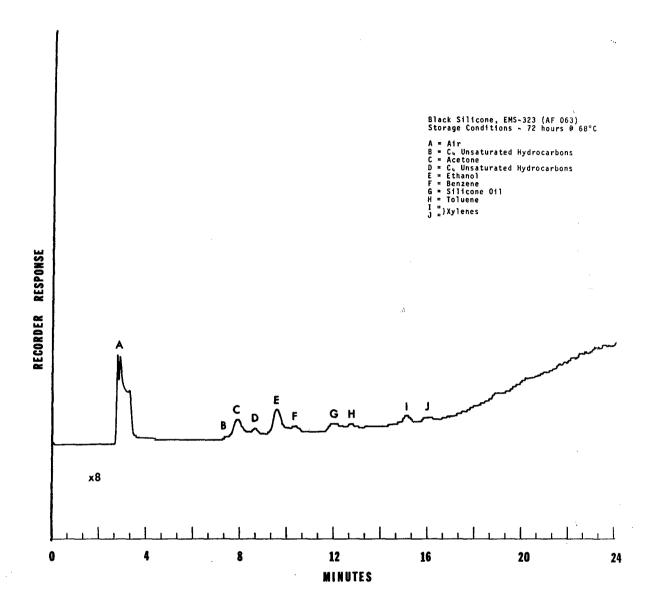


Figure 69. Gas Chromatogram of Gas-Off Products from Black Silicone, EMS-323 (AF 063) (72 hours @ 68°C).

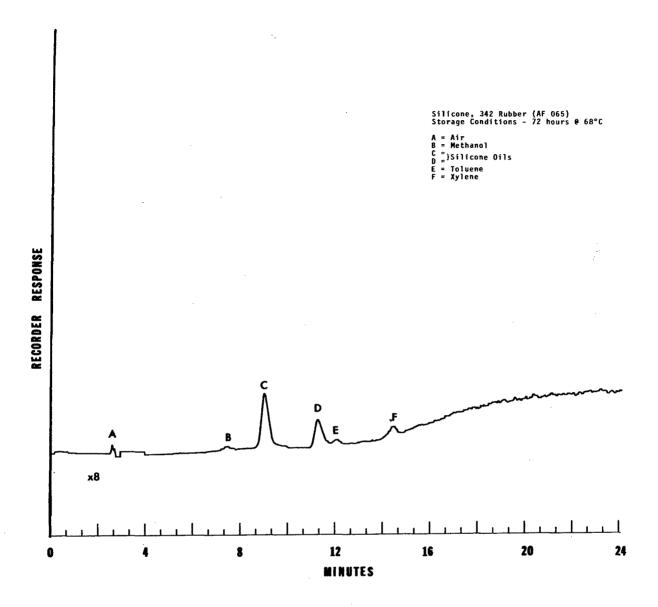


Figure 70. Gas Chromatogram of Gas-Off Products from Silicone, 342 Rubber (AF 065) (72 hours @ 68°C).

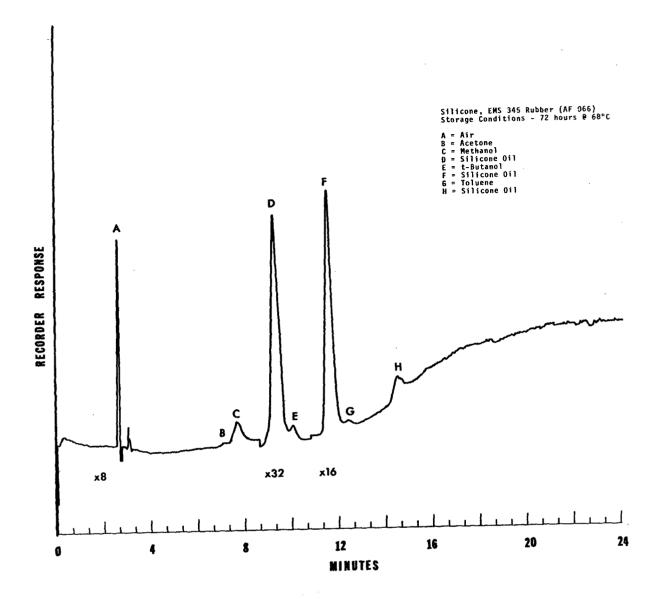


Figure 71. Gas Chromatogram of Gas-Off Products from Silicone, EMS 345 Rubber (AF 066) (72 hours @ 68°C).

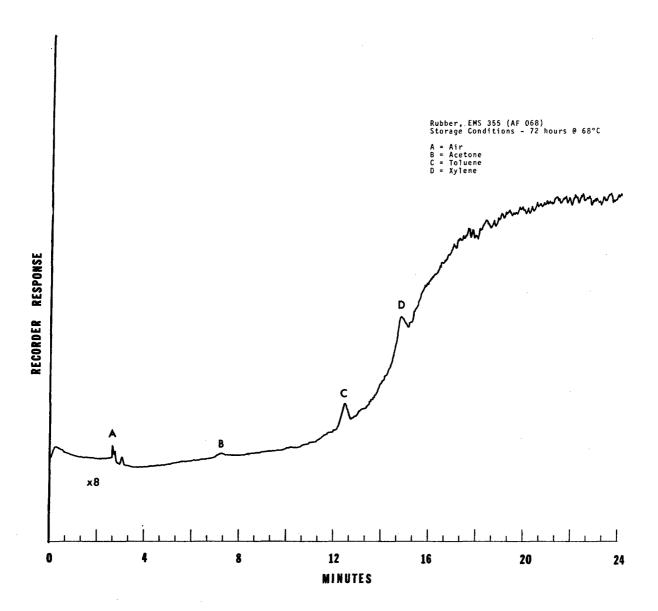


Figure 72. Gas Chromatogram of Gas-Off Products from Rubber, EMS 355 (AF 068) (72 hours @ 68°C).

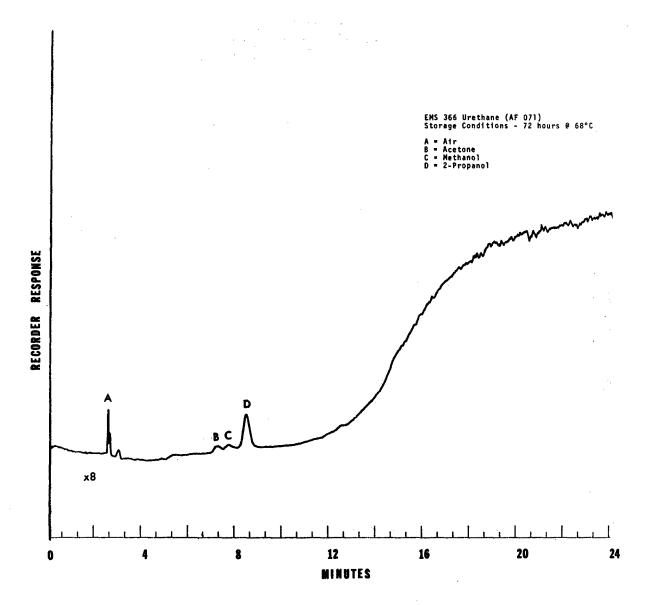


Figure 73. Gas Chromatogram of Gas-Off Products from EMS 366 Urethane (AF 071) (72 hours @ 68°C).

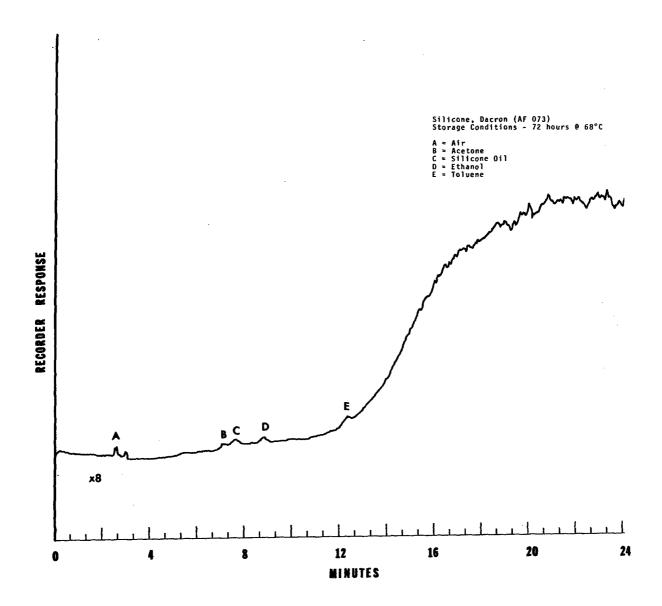


Figure 74. Gas Chromatogram of Gas-Off Products from Silicone, Dacron (AF 0.3) (72 hours @ 68°C).

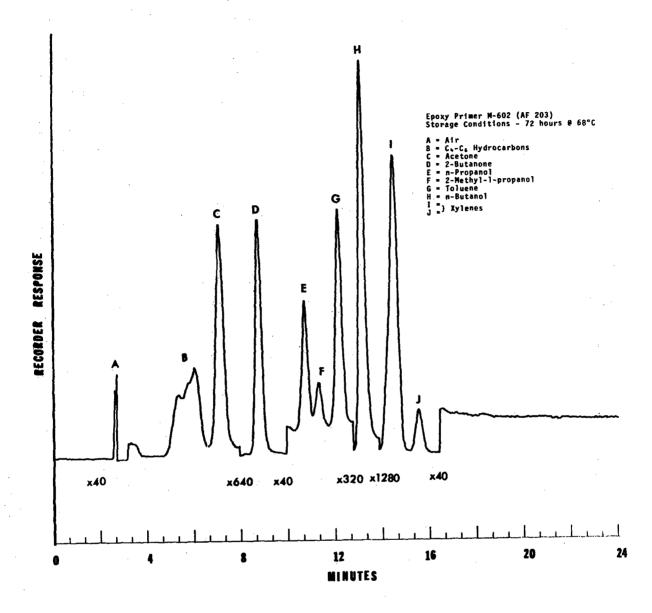


Figure 75. Gas Chromatogram of Gas-Off Products from Epoxy Primer M-602 (AF 203) (72 hours @ 68°C).

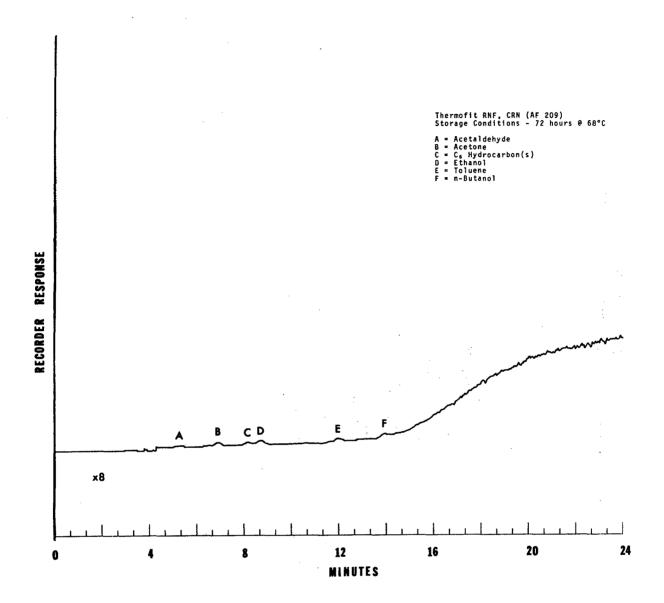


Figure 76. Gas Chromatogram of Gas-Off Products from Thermofit RNF, CRN (AF 209) (72 hours @ 68°C).

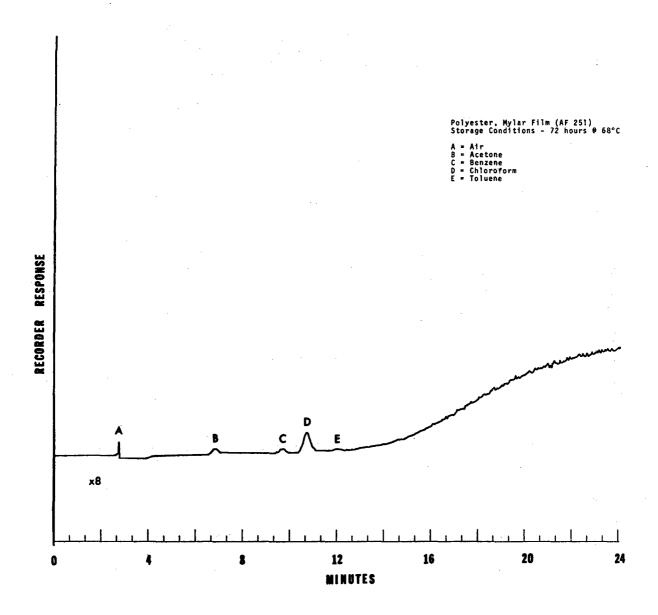


Figure 77. Gas Chromatogram of Gas-Off Products from Polyester, Mylar Film (AF 251) (72 hours @ 68°C).

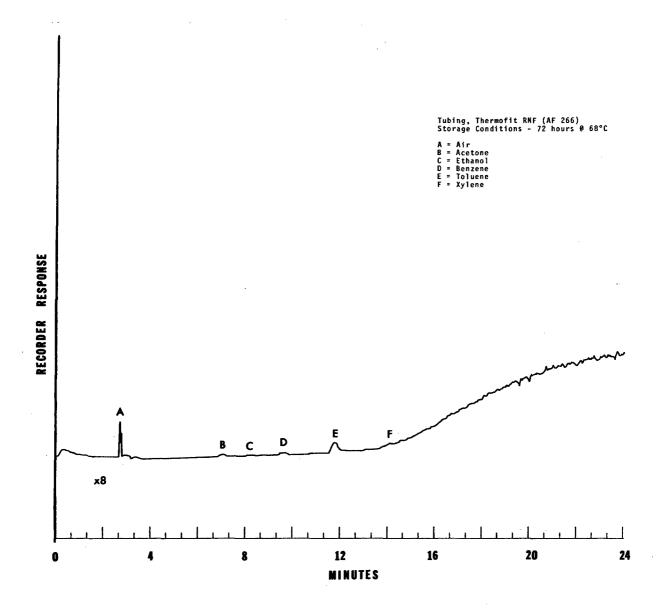


Figure 78. Gas Chromatogram of Gas-Off Products from Tubing, Thermofit RNF (AF 266) (72 hours @ 68°C).

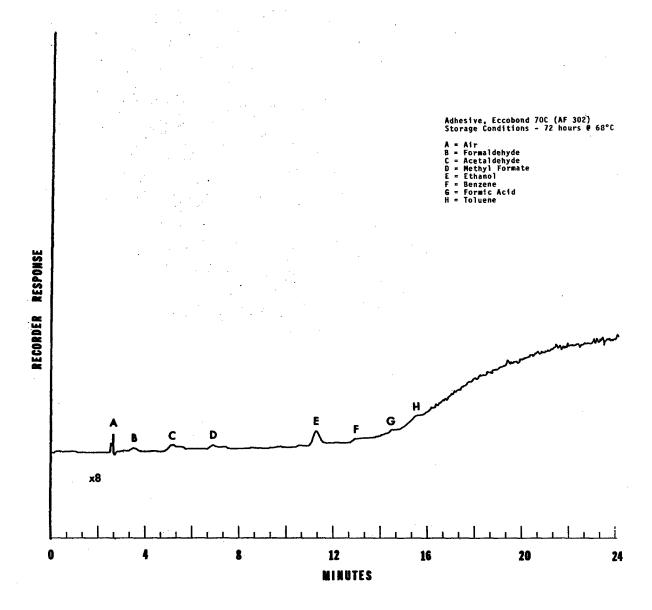


Figure 79. Gas Chromatogram of Gas-Off Products from Adhesive, Eccobond 70C (AF 302) (72 hours @ 68°C).

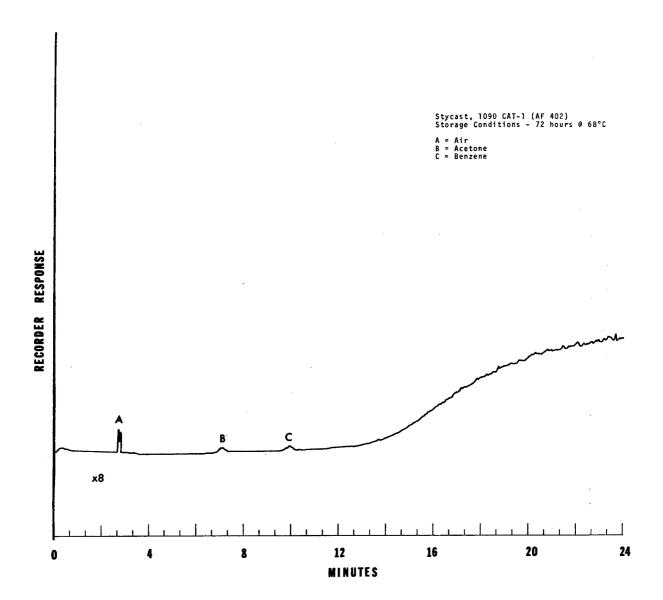


Figure 80. Gas Chromatogram of Gas-Off Products from Stycast, 1090 CAT-1 (AF 402) (72 hours @ 68°C).

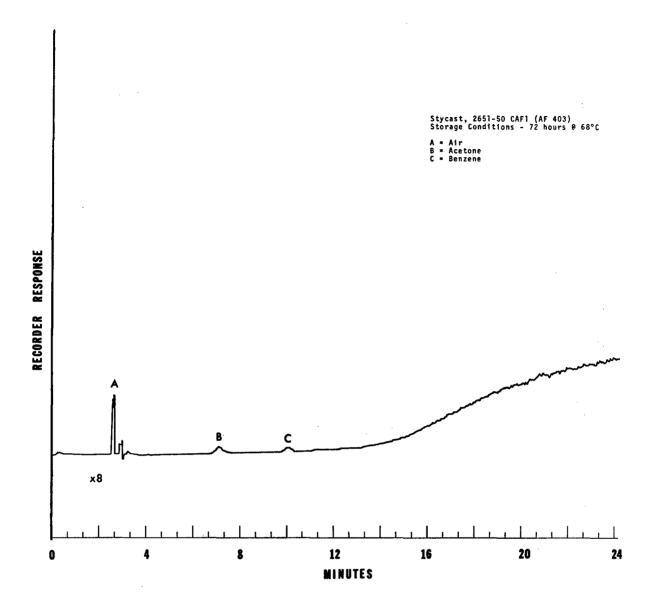


Figure 81. Gas Chromatogram of Gas-Off Products from Stycast, 2651-50 CAF1 (AF 403) (72 hours @ 68°C).

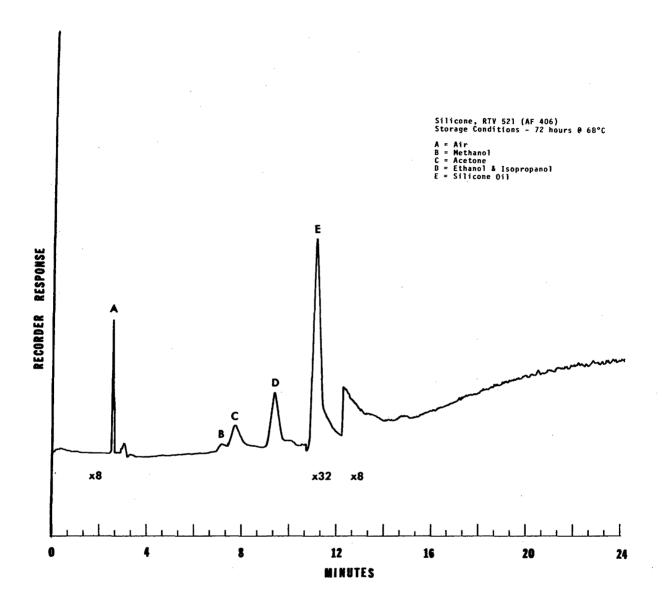


Figure 82. Gas Chromatogram of Gas-Off Products from Silicone, RTV 521 (AF 406) (72 hours @ 68°C).

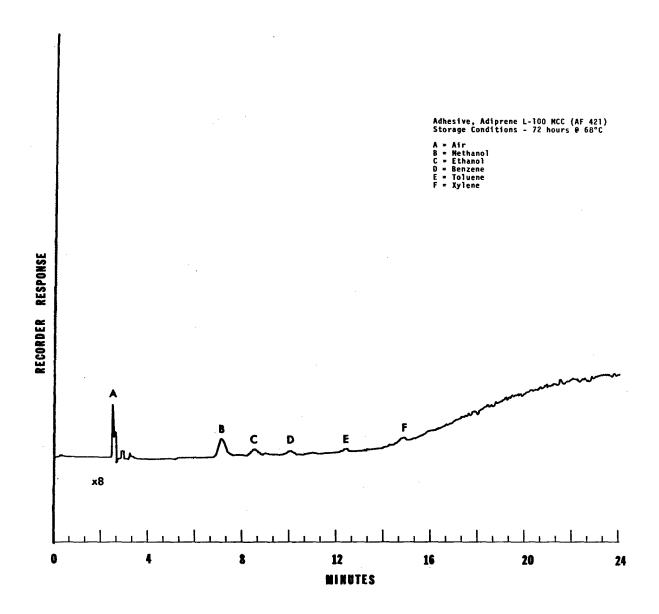


Figure 83. Gas Chromatogram of Gas-Off Products from Adhesive, Adiprene L-100 MCC (AF 421) (72 hours @ 68°C).

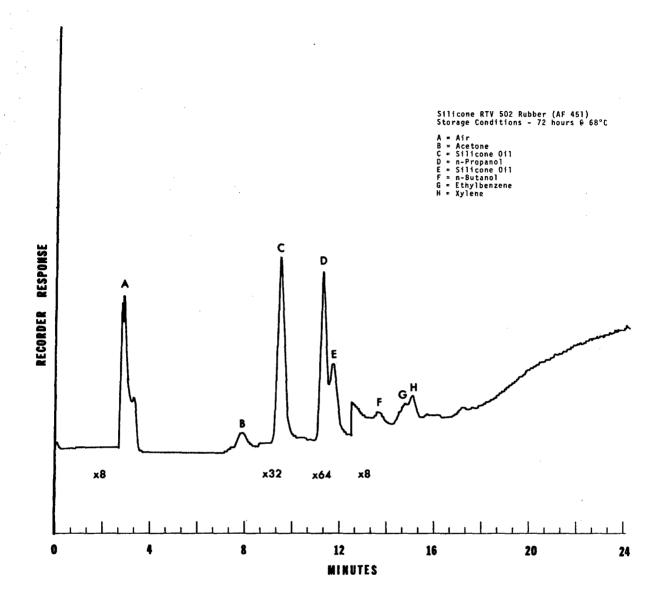


Figure 84. Gas Chromatogram of Gas-Off Products from Silicone RTV 502 Rubber (AF 451) (72 hours @ 68°C).

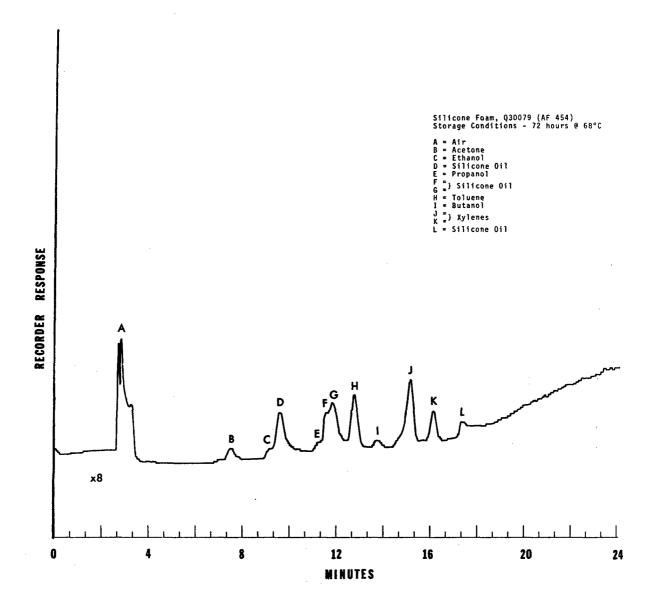


Figure 85. Gas Chromatogram of Gas-Off Products from Silicone Foam, Q30079 (AF 454) (72 hours @ 68°C).

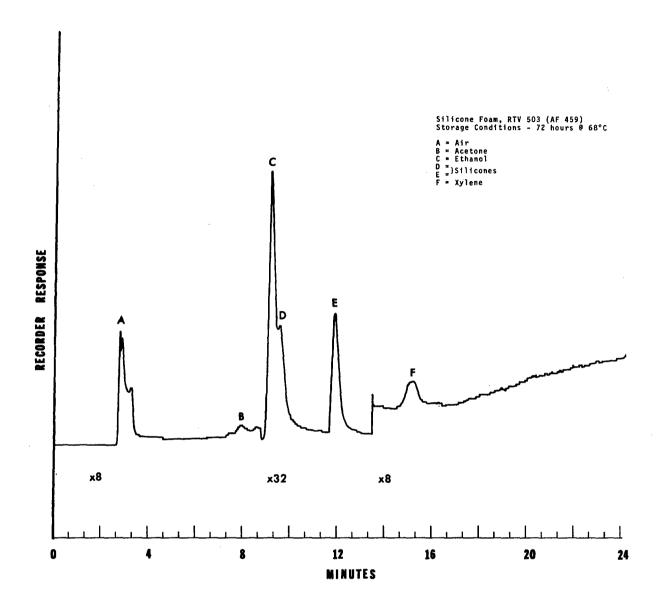


Figure 86. Gas Chromatogram of Gas-Off Products from Silicone Foam, RTV 503 (AF 459) (72 hours @ 68°C).

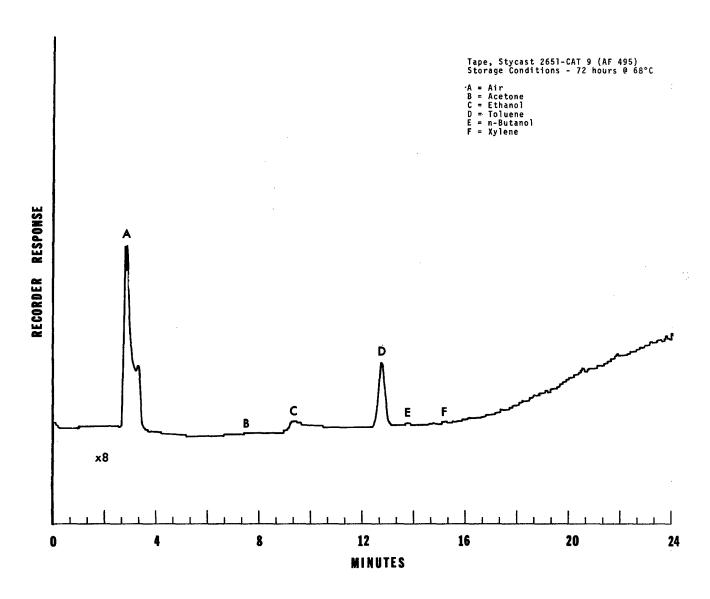


Figure 87. Gas Chromatogram of Gas-Off Products from Tape, Stycast 2651-CAT 9 (AF 495) (72 hours @ 68°C).

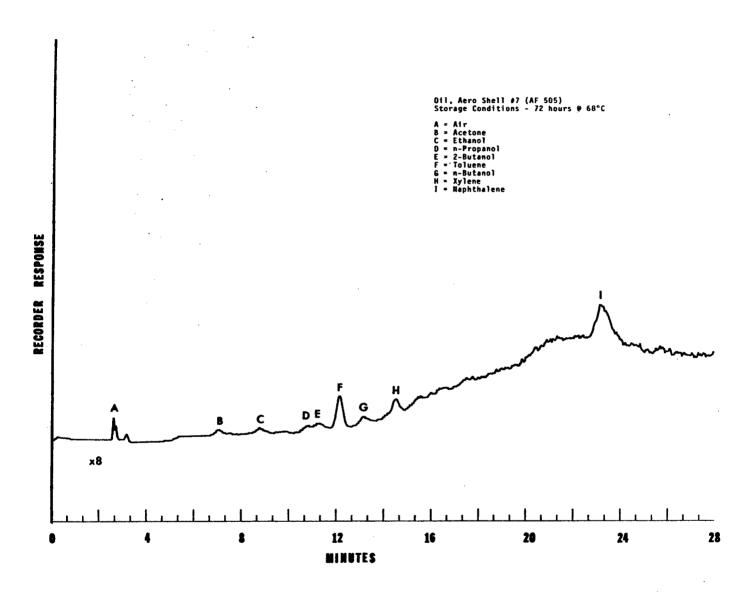


Figure 88. Gas Chromatogram of Gas-Off Products from Oil, Aero Shell #7 (AF 505) (72 hours @ 68°C).

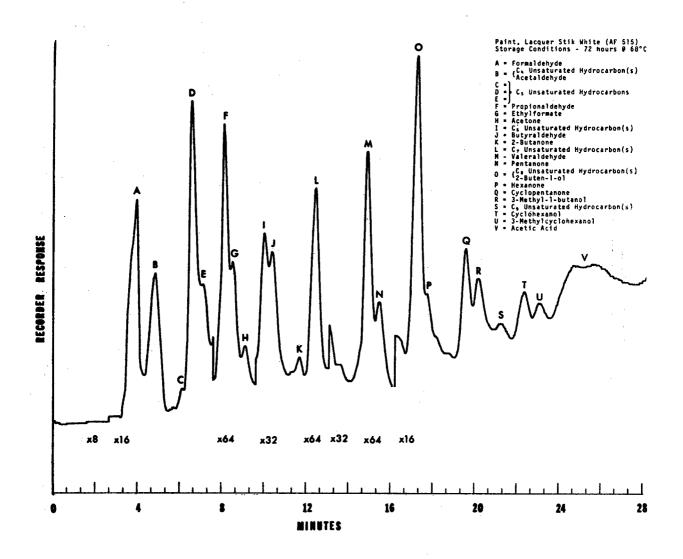


Figure 89. Gas Chromatogram of Gas-Off Products from Paint, Lacquer Stik White (AF 515) (72 hours @ 68°C).

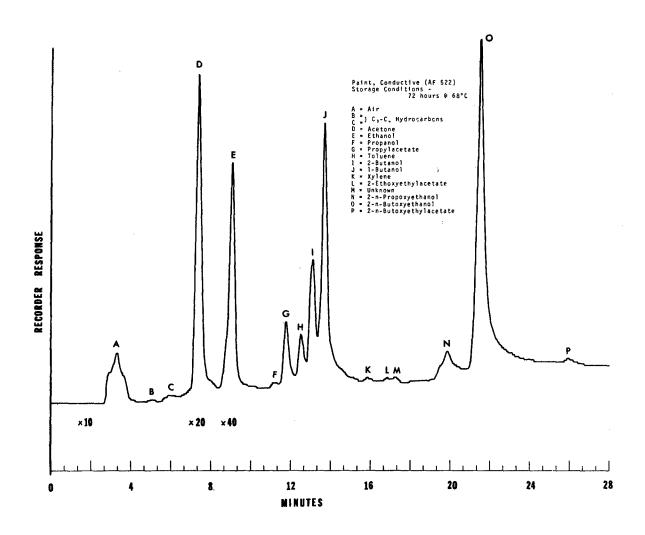


Figure 90. Gas Chromatogram of Gas-Off Products from Paint, Conductive (AF 522) (72 hours @ 68°C).

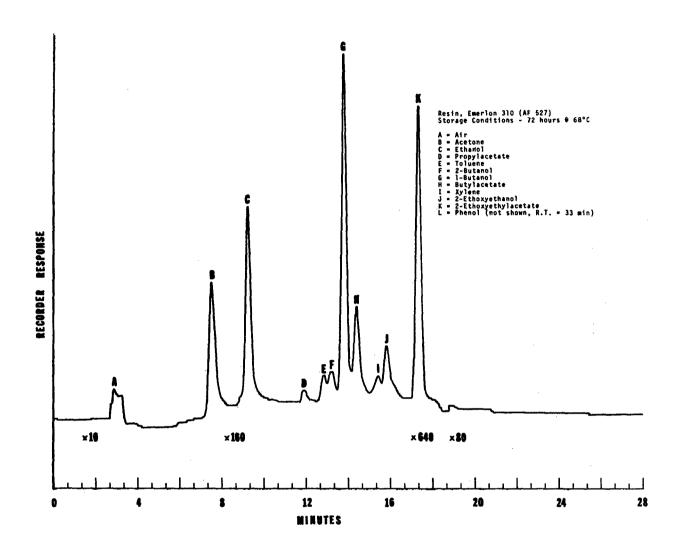


Figure 91. Gas Chromatogram of Gas-Off Products from Resin, Emerlon 310 (AF 527) (72 hours @ 68°C).

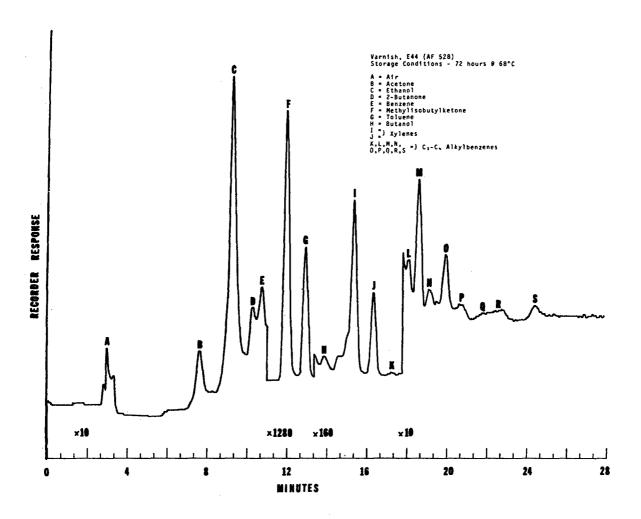


Figure 92. Gas Chromatogram of Gas-Off Products from Varnish, E44 (AF 528) (72 hours @ 68°C).

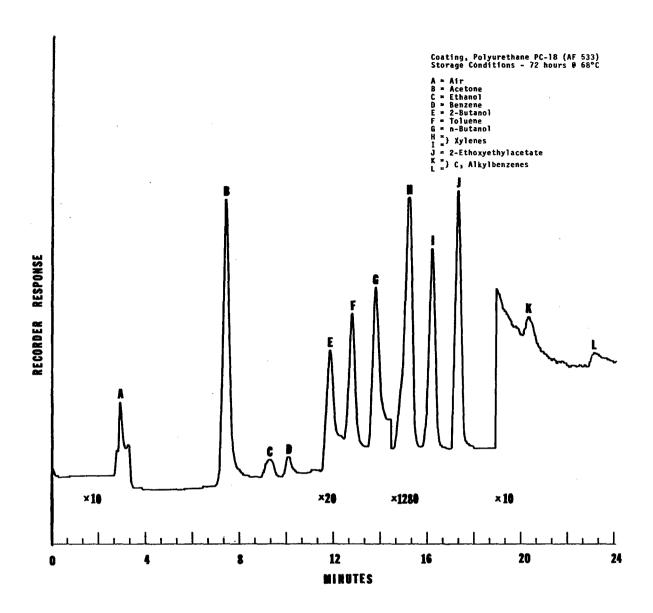


Figure 93. Gas Chromatogram of Gas-Off Products from Coating, Polyurethane PC-18 (AF 533) (72 hours @ 68°C).

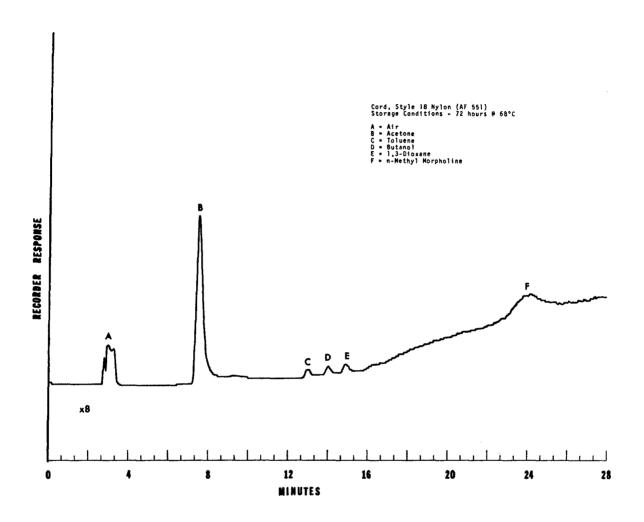


Figure 94. Gas Chromatogram of Gas-Off Products from Cord, Style 18 Nylon (AF 551) (72 hours @ 68°C).

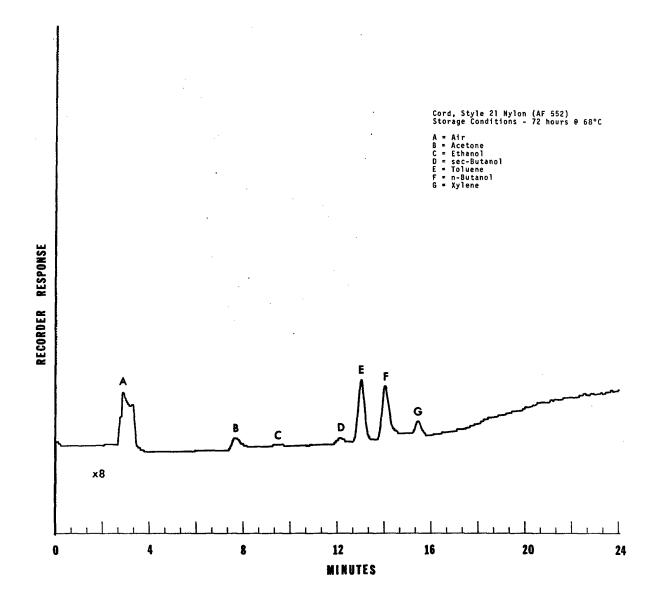


Figure 95. Gas Chromatogram of Gas-Off Products from Cord, Style 21 Nylon (AF 552) (72 hours @ 68°C).

APPENDIX IV

ANALYSES OF BIO-ENVIRONMENTAL ATMOSPHERES

AND SPECIAL ANALYSES

Table XLVI

SPECIAL ANALYSIS NO. 1

GAS-OFF PRODUCTS FROM HETRON POLYESTER RESIN

Components	Weight of Components After 30 Days at 25°C (mg/10 gm Candidate Material)		
Styrene	0.1		
Methylethylketone (or methylethylketone peroxide)*	0.4		
Divinylbenzene	0.02		
Xylene	0.005		
Acetone	0.01		
Ethanol	0.1		
Methane	0.02		
Carbon Monoxide	0.1		

^{*}Due to lack of data on the stability of MEK peroxide, it has not been established whether the peroxide decomposes during storage or during analysis.

Table XLVII

SPECIAL ANALYSES NOS. 2 AND 3

BIO-ENVIRONMENTAL SAMPLES

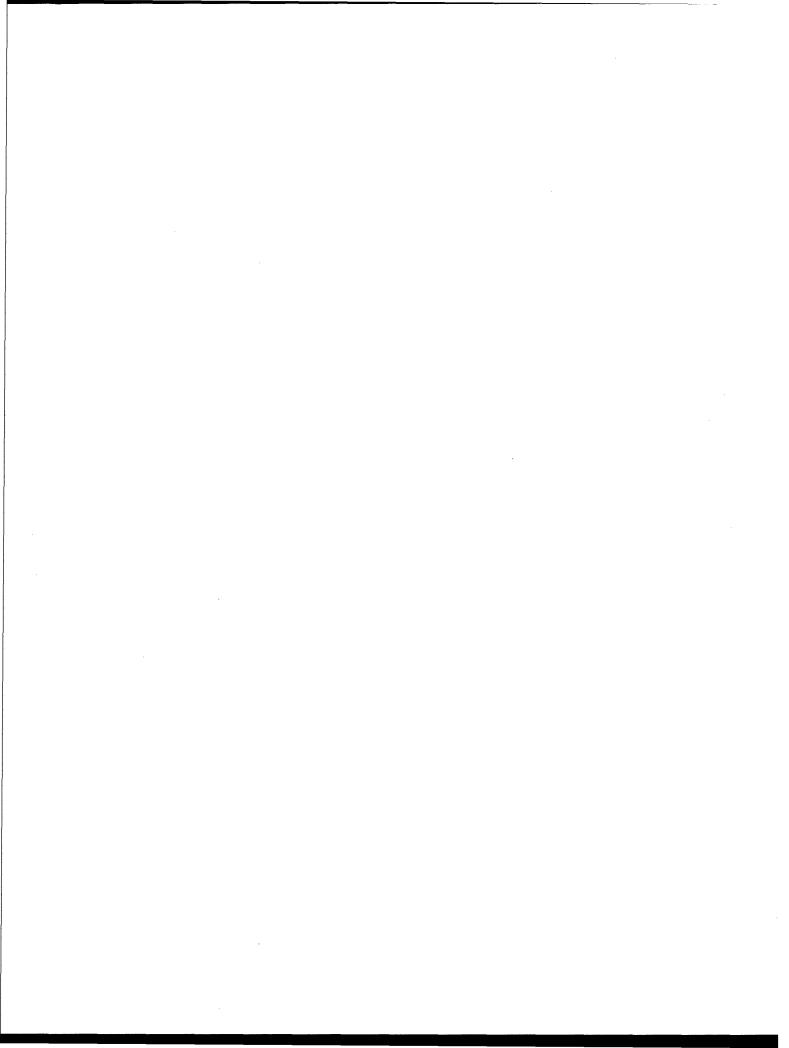
	No.2 Exhaust From Test Chamber	No.3 Room Air Outside Test Chamber
Carbon Dioxide	500 ppm	500 ppm
C ₁ -C ₂ Chlorinated Fluorocarbons*	20 ppm	15 ppm
C ₁ -C ₄ Alkylbenzenes	0.1 ppm	(not detected)
Carbon Monoxide	3 ppm	2 ppm
Methane	1-2 ppm	1-2 ppm

^{*}Similar to gas-off products observed from partially chlorinated fluorocarbon lubricants.

Table XLVIII

SPECIAL ANALYSES NOS. 4, 5, 6 AND 7 ANALYSIS OF FOUR BIO-ENVIRONMENTAL SPECIMENS

			Component	Level (mg/liter)
Cylinder	No.	1	Acetone Ethanol Benzene Trichloroethylene Toluene C ₁ -C ₂ Chlorofluorocarbons Methane Carbon Monoxide	0.02 <u>5</u> 0.03 0.0002 0.002 0.006 5.6 0.0008 0.0005
Cylinder	No.	2	Benzene Toluene Methane Carbon Monoxide	0.001 0.0007 0.004 0.0002
Cylinder	No.	3	Acetone Trichloroethylene Toluene Methane Carbon Monoxide	0.01 0.002 0.002 0.006 0.001
Cylinder	No.	4	Acetone Ethanol Benzene Toluene Methane Carbon Monoxide	0.001 0.0008 0.0005 0.002 0.002 0.003



Security Classification					
DOCUMENT CONT	=				
(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)					
1. ORIGINATING ACTIVITY (Corporate author) Monsanto Research Corporation	<u>j</u>	LASSIFIED			
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Fifty-three candidate materials for space call volatile gas-off and oxidation products. Test					
(a) preliminary screening by thermogravimetric analysis to determine weight loss during 24 hours at 25°C to 68°C in a nitrogen atmosphere at 5 psia.					
(b) storage tests at 68°C for 72 hours and at 25°C for 30 and 60 days in oxygen at 5 psia, followed by analyses of the chamber gases.					
The preliminary screening by measuring weight which exhibit weight losses between 0.001% falling within this range were studied further the individual components evolved from the outside this range were conditionally exclude thermogravimetric curves, gas chromatogram and quantities of individual components evolved. In addition to the gas-off experim spectrometric analyses were performed on 7 mental systems.	and 1.0%, exclusive in storage tests to decandidate material. The defrom further tests. s of volatile contaminated from the candidate ents, gas chromatogra	of water. Materials etermine the nature of hose materials falling Weight loss data, ants, and the nature e materials are phic and mass			

Security Classification LINK A LIŅK C LINK B KEY WORDS ROLE WT ROLE ROLE WT Space cabin candidate materials Volatile contaminant analyses Thermogravimetric analyses Mass spectrometry Gas chromatography Bio-environmental analyses